A PERSONAL PORTABLE ANALYTICAL KIT (PPAK) FOR THE ANALYSIS OF ORGANIC AND INORGANIC COMPOUNDS IN WATER AND SOIL

by

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ABSTRACT

The Personal Portable Analytical Kit has been developed for on-site analysis of chemical spills in soil or water in which a single known chemical is involved. This system uses colorimetric and titrimetric techniques for the analysis of chemicals from the Environment Canada spill priority list. From this list, the kit can quantify 67 contaminants in water and 53 contaminants in soil. All of the information required for the use of these test methods for the determination of concentrations of environmental contaminants is detailed in this report in conjunction with the Hach Water Analysis Handbook.

RÉSUMÉ

La trousse portative d'analyse a été conçue pour l'analyse sur le terrain de déversements de produits chimiques dans le sol ou dans l'eau, pour lesquels un seul produit chimique connu est en cause. Ce système fait appel à des techniques colorimétriques pour l'analyse de produits chimiques inscrits sur la liste prioritaire des déversements d'Environnement Canada. D'après cette liste, la trousse peut quantifier 67 contaminants dans l'eau et 53 contaminants dans le sol. Toutes les informations requises pour l'application de ces méthodes d'analyse pour la détermination des concentrations de contaminants dans l'environnement sont détaillés dans ce rapport, conformément au Manuel d'analyse de l'eau de Hach.

TABLE OF CONTENTS

	Page
Abstract	i
Contents	ii
Acknowledgements	vii
Introduction	1
The Personal Portable Analytical Kit	1
Colorimetric Methods	2
Titrimetric Methods	3
Conclusion	4
References	6
Section A - Methods	8
Acetic Acid	
Water and Wastewater	9
Acetic Anhydride	
Soils	11
Water and Wastewater	13
Acetone	
Soils	15
Water and Wastewater	18
Acrylonitrile	
Soils	20
Water and Wastewater	23
Adipic Acid	
Soils	26
Water and Wastewater	28
Aluminum Chloride	
Soils	30
Water and Wastewater	33
Aluminum Sulfate	
Soils	36

Ammonium Chloride
Soils 40
Water and Wastewater 43

Ammonium Nitrate
Soils 46
Water and Wastewater 48

Ammonium Phosphate
Water and Wastewater 50

Ammonium Sulfate
Soils 52

Benzene
Soils 55
Water and Wastewater 57

Benzoic Acid
Water and Wastewater 60

Boric Acid
Soils 62

1-Butanol
Soils 64
Water and Wastewater 66

iso-Butyraldehyde
Soils 68
Water and Wastewater 71

n-Butyraldehyde
Soils 73
Water and Wastewater 76

Calcium Chloride
Soils 79
Water and Wastewater 82

Calcium Hydroxide
Water and Wastewater 85

Calcium Oxide
Water and Wastewater 87

Calcium Phosphate
Water and Wastewater 89

Cumene
Soils 91
Water and Wastewater 93

Ethanol
Soils 90
Water and Wastewater 97

Ethanolamine
Water and Wastewater 99

Ethyl Benzene
Soils 102
Water and Wastewater 104

Ethylene Glycol
Soils 106
Water and Wastewater 109

Ferric Chloride
Soils 112
Water and Wastewater 115

Formic Acid
Water and Wastewater 118

Hydrochloric Acid
Soils 120
Water and Wastewater - Method 1 123
- Method 2 126

Hydrogen Peroxide
Water and Wastewater - Method 1 128
- Method 2 131

Isopropanol
Soils 134
Water and Wastewater 136

Maleic Anhydride
Soils 138
Water and Wastewater 140

Methanol
Soils 142
Water and Wastewater 144

Methyl Ethyl Ketone
Soils 146
Water and Wastewater 149

Methyl Isobutyl Ketone
Soils 152
Water and Wastewater 155

Morpholine
Soils 158
Water and Wastewater 161

Nitric Acid
Water and Wastewater 164

Nitrilotriacetic Acid
Soils 166
Water and Wastewater 168

Phosphoric Acid
Water and Wastewater - Method 1 170
- Method 2 172

Phthalic Anhydride
Soils 174

Potassium Chloride
Soils 176
Water and Wastewater 179

Potassium Hydroxide
Water and Wastewater 182

Potassium Sulfate
Soils 184

Propylene Glycol
Soils 187
Water and Wastewater 190

Sodium Carbonate
Water and Wastewater 193

Sodium Chloride
Soils 195
Water and Wastewater 198

Sodium Hydroxide
Water and Wastewater 201

Sodium Phosphate
Water and Wastewater 203

Sodium Sulfate
Soils 205

Sulfuric Acid
Water and Wastewater 209

p-Terphenyl
Soils 211

Toluene
Soils 213
Water and Wastewater 216

Urea
Soils 219
Water and Wastewater 221

Vinyl Acetate
Soils 223

Xylenes
Soils 226
Water and Wastewater 229

Zinc Chloride
Soils 232
Water and Wastewater 235

Zinc Sulfate
Soils 238

Section B - Calibration Data 242

Section C - Applicable Meterscale Overlays 251

Section D - Summary of PPAK Capabilities 268

Section E - PPAK Implementation Requirements 272

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Introduction

The person-portable analytical system (Level II) consists of instruments intended for use at spill sites where the identity of the hazardous material is known. These instruments are effective in spill response for measuring an ambient concentration, determining the gross extent of contamination, identifying contaminant plumes and implementing effective countermeasures (Bobra, 1986). The contaminants of interest are the chemicals on the Environmental Protection Service spill priority list. The instrumentation provides rapid and accurate analysis of air, water and/or soil samples for one or more of these contaminants. The methods for use with this instrumentation are simple and straightforward. This allows use by response teams without extensive training. One of the instruments selected by Environment Canada for modification and inclusion into the Level II system is the Hach Company Direct Reading Engineer's Laboratory Model DR-EL/4 (Hach kit).

The commercially available instrument is capable of detecting all of the inorganic chemicals from Environment Canada's spill priority list in contaminated water systems, with the exception of hydrogen peroxide and titanium dioxide, by identifying a specific ion in solution. In most cases, however, the kit is not calibrated to quantify specific compounds. The only organic compound which can be analyzed by this kit is phenol. This portable laboratory contains a spectrophotometer, pH meter, conductivity meter, digital titrator, basic glassware, and pre-packaged reagents. Also included in the kit is a well written and comprehensive manual.

The Personal Portable Analytical Kit (PPAK)

Work on expansion of the Hach kit had focused on developing procedures and methodologies for the analysis of spill priority chemicals in soil and water. The result has been the Personal Portable Analytical Kit, or PPAK. This modified kit incorporates all of the features of the commercially available Hach kit as well as the additions developed in this project.

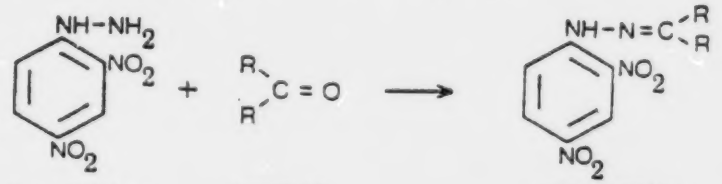
Initially, an extensive literature search was conducted, and compound-specific tests were located and evaluated in regard to the Level II concept of portability. Those methods that passed preliminary evaluations were tested and/or modified for inclusion into the PPAK. Test criteria for acceptance included accuracy, reproducibility, sensitivity, absence of interferences, range of detection, and field portability. Laboratory work on water analysis focused on the extraction and testing of various contaminants in de-ionized water. Laboratory work performed on soils included the extraction of contaminants from two distinct

soil types; sand and clay-based. Analytical techniques used in conjunction with the PPAK are either colorimetric or titrimetric methods.

Colorimetric Methods

The PPAK employs a large range of colorimetric tests for the analysis of specific compounds in water. Calibrated meterscale overlays are used in conjunction with a wavelength setting on the spectrophotometer for each specific test. These overlays eliminate the need to prepare standards when calibrating the spectrophotometer and provides the spill responder with direct concentration readings.

The 2,4-Dinitrophenylhydrazine (2,4-DNPH) test is an example of a colorimetric method used in the PPAK. This test is capable of detecting three ketones (acetone, methyl ethyl ketone, and methyl isobutyl ketone) and three aldehydes (formaldehyde, n-butyraldehyde, and iso-butyraldehyde) from Environment Canada's spills list. The reaction principle is illustrated below.



Potassium Hydroxide is added to produce a wine-red colour, presumably due to a resonating quinodal ion (Critchfield, 1963).

The intensity of the colour is then measured by the spectrophotometer in the kit at 480 nm.

Repetitive testing and calibrations of individual compound concentrations against the spectrophotometer readings enabled the test data to be converted into a form usable by the spill responder. Figure 1 is an example of the meterscale overlay for the 2,4-Dinitrophenylhydrazine test for Acetone in water.

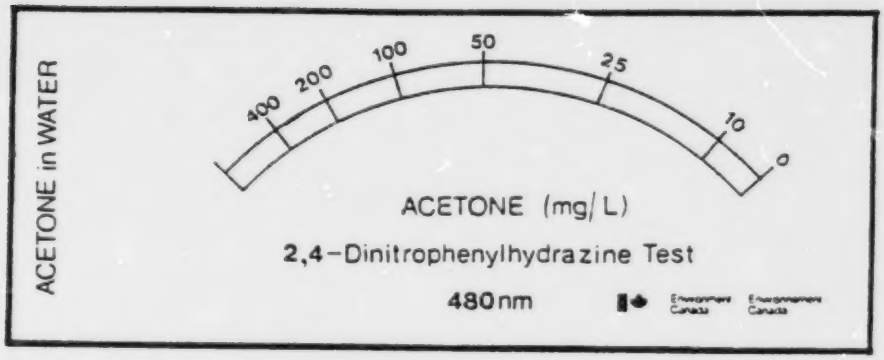


Figure 1. Meterscale Overlay for Acetone in Water.

By using the appropriate colorimetric test and meterscale overlay, contaminant concentrations can be read directly from the spectrophotometer in units of mg/L of water or mg/kg of soil. Colorimetric methodologies which have been tested and proven suitable for inclusion into the PPAK are found in Section A of this report. A total of 42 specific contaminants can be determined colorimetrically with these tests. The tabulated calibration data is contained in Section B and the meterscale overlays are presented in Section C.

Titrimetric Methods

The PPAK also employs a number of titrimetric methods for the analyses of specific compounds. Addition of measured amounts of titrant to the treated contaminated sample causes a visible colour change, signifying the titrimetric endpoint. Contaminant concentrations are determined through simple equations and calibration curves. Prior to their inclusion into the PPAK, potential methods were screened. Two of the selection objectives were for tests capable of detecting more than one compound and for those which enabled simple and effective calculation of contaminant concentrations from calibration data.

The Periodic Acid (Redox) Method for 1,2 Glycols is one such example of a titrimetric method incorporated into the PPAK. In this test, periodic acid is used for the selective oxidation of organic compounds with hydroxyl groups on adjacent carbon atoms (Critchfield, 1963). Ethylene glycol and propylene glycol are

two prime candidates. Periodate oxidation of hydroxylamines, compounds having hydroxyl and amino groups on adjacent atoms, may also occur. The reaction principle, using ethylene glycol, is illustrated.

In the Periodic Acid method for ethylene glycol, two moles of formaldehyde are formed when a measured excess of periodic acid is added. The oxidation reaction is complete in 20-25 minutes at room temperature. The reaction mixture is rendered slightly alkaline with saturated sodium bicarbonate, an excess of potassium is introduced, and the iodine liberated by the excess periodate is then consumed by a measured excess of sodium arsenite. The As(III) is then titrated with a standard iodine solution. Starch is used as the indicator.

Section A contains methodologies for 72 titrimetric tests. The method of calculation of contaminant concentration is the same for all of the included titrimetric tests. Initially, the calibration data was plotted graphically and a calibration curve was determined. From the curve, a linear equation was computed to enable generalized usage for variable titrant concentrations. This equation has been set up as follows:

Contaminant Concentration Equation -

Concentration (mg/kg) =[# mL's of titrant] x
[normality of titrant] x
[Contaminant Factor]

The Contaminant Factor is a numerical multiplier. Each contaminant has it's own unique factor. The Contaminant Factors are included in the calculation segment of each titrimetric methodology in Section A.

Conclusions

The modification of the Hach kit has increased the number of testing procedures of chemicals from Environment Canada's spills priority list. The capacity of the kit for use in spill situations has been expanded from 6 specific analytical tests in

water to 67 tests, with an additional 53 tests in soil. In order to support the addition of these tests, a total of 32 reagents have been added. Comprehensive testing and calibration of colorimetric methods has resulted in the development of compound-specific meterscale overlays. Similarly, the results of the titrimetric methods have led to the computation of contaminant factors. The culmination of these efforts is presented in Section A as a manual for use by responders to chemical spill situations.

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SECTION A

ACETIC ACID IN WATER AND WASTEWATER

SODIUM HYDROXIDE TITRATION METHOD

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0 - 10000 mg/L

Common Synonyms: Glacial Acetic Acid, Ethanoic Acid, Methane Carboxylic Acid, Vinegar Acid

HAZARDOUS DATA

Human Health
Symptoms: Contact - solutions are corrosive and can cause severe burns to tissue. Inhalation - concentrated vapours can cause coughing, chest pain, irritation of nose and throat, nausea. Ingestion - burning sensation, nausea, and vomiting.
Toxicology: Corrosive upon inhalation, ingestion and contact.
TLV - 10 ppm; 25 mg/cubic meter

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 50 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
1 x 25 mL Burette

REQUIRED REAGENTS

Phenolphthalein indicator solution
0.02N Sodium Hydroxide solution
*Note: All required reagents supplied by Hach company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the

flask TEST.

3. Fill the 0 - 25 mL burette with 0.02N Sodium Hydroxide solution and record the initial burette reading.

4. Add 5 drops of phenolphthalein solution to each flask.

5. Titrate the BLANK water sample against the sodium hydroxide solution; add slowly, with continuous swirling until a pink colour appears for more than 30 seconds.

6. Record the final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Acetic Acid by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 23.9]

ACETIC ANHYDRIDE IN SOIL

HYDROLYSIS AND SODIUM HYDROXIDE TITRATION

Reference: Smith, D.M., Bryant, W.M.D., J. Am. Chem. Soc., 58, 2452, 1936.

Range: 500 - 5000 mg/kg

Common Synonyms: Acetic Oxide, Acetyl Oxide, Acetyl Ether

HAZARDOUS DATA

Readily Combustible.

Human Health
Symptoms: Inhalation - coughing, chest pain, nausea, vomiting.
Ingestion - burning sensation, nausea, vomiting, convulsions.
Contact - skin and eye irritant, burns.
Toxicology: Corrosive by all routes.

Environment:
Aquatic Toxicity Rating - 10 to 100/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 125 Erlenmeyer flasks
- 2 x funnel
- filter paper
- 2 x 150 mL beaker
- 50 mL graduated cylinder
- 2 x magnetic stirring bars
- magnetic stirrer
- 25 mL buret

REQUIRED REAGENTS

- 1:1 Pyridine/Water solution
- 0.02N Sodium Hydroxide
- Thymol blue indicator solution

Preparation of 1:1 pyridine/water solution -

Combine 75 mL of deionized water with 75 mL of pyridine in a 500 mL bottle with a screw-top lid.

Preparation of Thymol blue indicator solution -

Dissolve 0.4 grams of Thymol blue in 100 mL of deionized water and store in a 150 mL plastic bottle with lid.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of pyridine/water solution to each flask. Mix well for 5 minutes.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Put a magnetic stirring bar in each beaker. Add 6 drops of Thymol blue indicator to each sample.

6. Fill the buret with 0.02N Sodium Hydroxide solution. Record the initial buret reading. Place the BLANK sample on the magnetic stirrer and turn stirrer on to low speed. Titrate the BLANK sample with Sodium Hydroxide until the solution changes colour from yellow to blue-green. Record the final buret reading.

7. Refill the buret. Record initial buret reading. Titrate the TEST sample as above. Record final buret reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Acetic Anhydride by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x 131]

ACETIC ANHYDRIDE IN WATER

SODIUM HYDROXIDE TITRATION

Reference: Smith, D.M., Bryant, W.M.D., J. Am. Chem. Soc., 58, 2452, 1936.

Range: 10 - 2000 mg/L

Common Synonyms: Acetic Oxide, Acetyl Oxide, Acetyl Ether

HAZARDOUS DATA

Readily Combustible.

Human Health
Symptoms: Inhalation - coughing, chest pain, nausea, vomiting.
Ingestion - burning sensation, nausea, vomiting, convulsions.
Contact - skin and eye irritant, burns.
Toxicology: Corrosive by all routes.

Environment:
Aquatic Toxicity Rating - 10 to 100/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer flasks
50 mL graduated cylinder
2 x magnetic stirring bars
magnetic stirrer
25 mL buret

REQUIRED REAGENTS

0.02N Sodium Hydroxide
Phenolphthalein indicator solution

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the

flask TEST.

3. Put a magnetic stirring bar in each flask. Add 6 drops of Phenolphthalein indicator to each sample.

4. Fill the buret with 0.02N Sodium Hydroxide solution. Record the initial buret reading. Place the BLANK sample on the magnetic stirrer and turn stirrer on to low speed. Titrate the BLANK sample with Sodium Hydroxide until pink colour remains for at least 1 minute. Record the final buret reading.

5. Refill the buret. Record initial buret reading. Titrate the TEST sample as above. Record final buret reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Acetic Anhydride by solving the following equation:

$$\text{Concentration (mg/L)} = [\ (\text{adjusted titrant volume}) \times 20\ ]$$

ACETONE IN SOIL

2,4-DINITROPHENYLHYDRAZINE METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Permagon Press, 1963.

Range: 0 - 1200 mg/kg

Common Synonyms: Dimethylketone, 2-Propanone, Methyl Ketone

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin- slight reddening and dryness; eyes- redness and irritation. Inhalation - coughing, headache, nausea, dizziness, narcosis. Ingestion - sore throat, headache, dizziness, narcosis, coma.
Toxicology: Only slightly toxic upon contact, inhalation and ingestion.
TLV - 750 ppm; 1780 mg/cubic meter
Short-term Inhalation Limits - 1000 ppm; 2375 mg/cubic meter

Environment: Prevent entry into waterways and intakes. Harmful to aquatic life. Fish toxicity: 14250 ppm/24h/sunfish/killed /tapwater; BOD: 38 to 81 %, 5 days.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
2 x 100 mL Mixing Cylinders
5 x 10 mL Disposable Pipets
2 x 150 mL Beakers
1 x 100 mL Volumetric Flask
Timing Device
Whatman #1 Filter Paper
2 x Long Stemmed Funnels
Balance

REQUIRED REAGENTS

Pyridine Stabilizer
33% Potassium Hydroxide Solution
2,4-Dinitrophenylhydrazine Solution

Preparation of Pyridine Stabilizer

Add 100 mL of deionized water to 400 mL of pyridine. Mix thoroughly. Store in a brown glass bottle.

Preparation of 33% Potassium Hydroxide Solution

Dissolve 33.0 g of potassium hydroxide in a 150 mL beaker containing 100 mL of methanol.

Preparation of 2,4-Dinitrophenylhydrazine solution

Dissolve 0.100 g of 2,4-Dinitrophenylhydrazine in a 100 mL beaker containing 50 mL of methanol and 4 mL of concentrated hydrochloric acid. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Transfer 50 mL of each filtrate into separate mixing cylinders.

6. Pipet 1.0 mL of the 2,4-Dinitrophenylhydrazine solution into each mixing cylinder. Stopper and shake gently. Wait exactly 20 minutes.

7. Pipet 1.0 mL of the 33% potassium hydroxide solution into each mixing cylinder. Then add 5 mL of pyridine stabilizer to each and stopper. Mix well and wait 2 minutes for colour stabilization.

8. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

9. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds

to the zero mark on the card.

10. Replace the Zero Scale card with the ACETONE in SOIL card.

11. Fill one cuvette to the 25 mL mark from the cylinder labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

12. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

13. Fill the other cuvette to the 25 mL mark from the cylinder labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the acetone concentration from the card and record this result.

ACETONE IN WATER

2,4-DINITROPHENYLHYDRAZINE METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Permagon Press, 1963.

Range: 0 - 400 mg/L

Common Synonyms: Dimethylketone, 2-Propanone, Methyl Ketone

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin- slight reddening and dryness; eyes- redness and irritation. Inhalation - coughing, headache, nausea, dizziness, narcosis. Ingestion - sore throat, headache, dizziness, narcosis, coma.
Toxicology: Only slightly toxic upon contact, inhalation and ingestion.
TLV - 750 ppm; 1780 mg/cubic meter
Short-term Inhalation Limits - 1000 ppm; 2375 mg/cubic meter

Environment: Prevent entry into waterways and intakes. Harmful to aquatic life. Fish toxicity: 14250 ppm/24h/sunfish/killed /tapwater; BOD: 38 to 81 %, 5 days.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
5 x 10 mL Disposable Pipets
2 x 150 mL Beakers
1 x 100 mL Volumetric Flask
Timing Device

REQUIRED REAGENTS

Pyridine Stabilizer
33% Potassium Hydroxide Solution
2,4-Dinitrophenylhydrazine Solution

Preparation of Pyridine Stabilizer

Add 100 mL of deionized water to 400 mL of pyridine. Mix thoroughly. Store in a brown glass bottle.

Preparation of 33% Potassium Hydroxide Solution

Dissolve 33.0 g of potassium hydroxide in a 150 mL beaker containing 100 mL of methanol.

Preparation of 2,4-Dinitrophenylhydrazine solution

Dissolve 0.100 g of 2,4-Dinitrophenylhydrazine in a 100 mL beaker containing 50 mL of methanol and 4 mL of concentrated hydrochloric acid. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. From an uncontaminated source in the area obtain a 100 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 100 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Pipet 2.0 mL of the 2,4-Dinitrophenylhydrazine solution into each flask. Stopper and shake gently. Wait exactly 20 minutes.

4. Pipet 2.0 mL of the 33% potassium hydroxide solution into each flask. Then add 10 mL of pyridine stabilizer to each and mix well and wait 2-3 minutes for colour stabilization.

5. Adjust the Wavelength Dial of the meter to read 480 nm.

6. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

7. Replace the Zero Scale card with the ACETONE in WATER card.

8. Fill one cuvette to the 25 mL mark from the flask labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

9. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

10. Fill the other cuvette to the 25 mL mark from the flask labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the acetone concentration from the card and record this result.

ACRYLONITRILE IN SOIL

PERCHLORIC ACID TITRIMETRIC METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Pergamon Press, New York, 1963.

Range: 500 - 4000 mg/kg

Common Synonyms: 2-propenenitrile, vinyl cyanide, cyanoethylene, Acritet, Fumigrain, Ventox

HAZARDOUS DATA

Explosive, flammable, Carcinogen!

Human Health:
Symptoms: Skin irritant, may cause dermatitis.
Toxicology: Highly toxic
TLV = 2 ppm

Environment:
Aquatic Toxicity Rating: 10 to 100 ppm/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 250 mL Erlenmeyer flasks
- 2 x magnetic stirring bars
- 50 mL graduated cylinder
- 10 mL graduated cylinder
- 25 mL buret
- Magnetic stirrer
- 2 x funnels
- filter paper
- 2 x 250 mL beaker

REQUIRED REAGENTS

- Methanol
- Morpholine
- Methyl Cellosolve
- Acetic Anhydride
- 0.1N Perchloric acid
- Thymol Blue / Xylene Cyanol FF indicator solution

Preparation of Perchloric acid solution, 0.1N -

Measure 10.7 mL of 70% perchloric acid and transfer to a 250 mL volumetric flask. Dilute to the mark with Methyl Cellosolve.

volumetric flask. Dilute to the mark with Methyl Cellosolve. Transfer 50 mL from this to another 250 mL volumetric flask. Dilute to the mark with Methyl Cellosolve. Use this to titrate.

Preparation of Thymol Blue/Xylene Cyanol FF indicator solution -

Dissolve 0.3 grams of thymol blue and 0.08 grams of xylene cyanol FF in 100 mL of dimethylformamide (DMF).

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of Methyl Cellosolve to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 250 mL beakers. Label the beakers to match the originating flask.

5. Put a magnetic stirring bar in each beaker.

6. Add 10 mL of Morpholine, using a clean 10 mL graduated cylinder, to each beaker. Then add 10 mL of Methanol to each beaker, shake well to mix and wait 5 minutes for reaction to proceed.

5. Add 50 mL of Methyl Cellosolve to each beaker and stir. Slowly add 20 mL of Acetic Anhydride to each beaker. Caution- reaction is endothermic and the beakers may get hot!

6. Allow beakers to cool for 1 minute, then add 1 mL of thymol blue / xylene cyanol FF indicator solution to each beaker.

7. Fill the buret with 0.5N Perchloric acid. Record the initial buret reading. Place the BLANK sample on the magnetic stirrer. Turn stirrer to low speed. Titrate the BLANK sample with Perchloric acid until the green colour has disappeared. Record final buret reading.

8. Refill the buret. Record initial buret reading. Titrate the TEST sample as above. Record final buret reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Acrylonitrile by solving the following equation:

$$\text{Concentration (mg/kg)} = [\ (\text{adjusted titrant volume}) \times (\text{titrant concentration}) \times 9200\ ]$$

where titrant concentration is 0.1 unless above Perchloric acid solution is not used.

ACRYLONITRILE IN WATER

PERCHLORIC ACID TITRIMETRIC METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Pergamon Press, New York, 1963.

Range: 100 - 2000 mg/L

Common Synonyms: 2-Propenenitrile, Vinyl Cyanide, Cyanoethylene, Acritet, Fumigrain, Ventox

HAZARDOUS DATA

Explosive, flammable, Carcinogen!

Human Health:
Symptoms: Skin irritant, may cause dermatitis.
Toxicology: Highly toxic
TLV = 2 ppm

Environment:
Aquatic Toxicity Rating: 10 to 100 ppm/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer flasks
2 x magnetic stirring bars
50 mL graduated cylinder
10 mL graduated cylinder
25 mL buret
Magnetic stirrer

REQUIRED REAGENTS

Methanol
Morpholine
Methyl Cellosolve
Acetic Anhydride
0.5N Perchloric acid
Thymol Blue / Xylene Cyanol FF indicator solution

Preparation of Perchloric acid solution, 0.5N -

Measure 10.7 mL of 70% perchloric acid and transfer to a 250 mL

volumetric flask. Dilute to the mark with Methyl Cellosolve.

Preparation of Thymol Blue/Xylene Cyanol FF indicator solution -

Dissolve 0.3 grams of thymol blue and 0.08 grams of xylene cyanol FF in 100 mL of dimethylformamide (DMF).

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Put a magnetic stirring bar in each flask.

4. Add 10 mL of Morpholine, using a clean 10 mL graduated cylinder, to each flask. Then add 10 mL of Methanol to each flask, shake well to mix and wait 5 minutes for reaction to proceed.

5. Add 50 mL of Methyl Cellosolve to each flask and stir. Slowly add 20 mL of Acetic Anhydride to each flask. Caution - reaction is endothermic and the flasks may get hot!

6. Allow flasks to cool for 1 minute, then add 1 mL of thymol blue / xylene cyanol FF indicator solution to each flask.

7. Fill the buret with 0.5N Perchloric acid. Record the initial buret reading. Place the BLANK sample on the magnetic stirrer. Turn stirrer to low speed. Titrate the BLANK sample with Perchloric acid until the green colour has disappeared. Record final buret reading.

8. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Acrylonitrile by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x (titrant concentration) x 1035]

where titrant concentration is 0.5 unless standard Perchloric acid solution is not used.

ADIPIC ACID IN SOIL

SODIUM HYDROXIDE TITRATION

Reference: Smith, D.M., Bryant, W.M.D., J. Am. Chem. Soc., 58, 2452, 1936.

Range: 200 - 4000 mg/kg

Common Synonyms: Hexanedioc Acid, 1,4-Butanedicarboxylic Acid, Adipinic Acid.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation causing sneezing and coughing. Ingestion - sore throat, abdominal pain. Contact - eyes- irritation; skin - pronounced drying effect.
Toxicology: Slightly toxic on acute exposure.

Environment:
Aquatic Toxicity Rating - 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
2 x 125 Erlenmeyer flasks
2 x funnel
filter paper
2 x 150 mL beaker
50 mL graduated cylinder
2 x magnetic stirring bars
magnetic stirrer
25 mL buret

REQUIRED REAGENTS

0.02N Sodium Hydroxide
Thymol blue indicator solution

Preparation of Thymol blue indicator solution -

Dissolve 0.4 grams of Thymol blue in 100 mL of deionized water and store in a 150 mL plastic bottle with lid.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of deionized water to each flask. Mix well for 5 minutes.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Put a magnetic stirring bar in each beaker. Add 0.5 mL of Thymol blue indicator to each sample.

6. Fill the buret with 0.02N Sodium Hydroxide solution. Record the initial buret reading. Place the BLANK sample on the magnetic stirrer and turn stirrer on to low speed. Titrate the BLANK sample with Sodium Hydroxide until the solution changes colour from yellow to blue-green. Record the final buret reading.

7. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Adipic Acid by solving the following equation:

$$\text{Concentration (mg/kg)} = [\ (\text{adjusted titrant volume}) \times 250\ ]$$

ADIPIC ACID IN WATER

SODIUM HYDROXIDE TITRATION METHOD

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0 - 10000 mg/L

Common Synonyms: 1,4-Butanedicarboxylic Acid, Hexanedioic Acid, Adipinic Acid

HAZARDOUS DATA

Human Health
Symptoms: Contact(liquid) - eyes- irritation; skin - pronounced drying effect. Inhalation(dust, vapours) - irritation of mucous membranes causing sneezing and coughing. Ingestion - sore throat and abdominal pain.
Toxicology: Relatively non-toxic

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 50 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
1 x 25 mL Burette

REQUIRED REAGENTS

Phenolphthalein indicator solution
0.02N Sodium Hydroxide solution
*Note: All required reagents supplied by Hach company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Fill the 0 - 25 mL burette with 0.02N Sodium Hydroxide

solution and record the initial burette reading.

4. Add 6 drops of phenolphthalein solution to each flask.

5. Titrate the BLANK water sample against the sodium hydroxide solution; add slowly, with continuous swirling until a pink colour appears for more than 30 seconds.

6. Record the final burette reading.

7. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Adipic Acid by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 27.3]

ALUMINUM CHLORIDE IN SOILS

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/kg

Common Synonyms: Anhydrous Trichloroaluminum

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - sore throat, coughing, laboured breathing. Ingestion - burning sensation, stomach cramps, dizziness, diarrhea, shock, convulsions, coma. Contact - eyes- redness, pain, blurred vision; skin- redness, pain.
Toxicology: Moderately toxic, corrosive by ingestion and contact.
TLV - 5 ppm; 7 mg/cubic meter (as HCl)

EXPERIMENTAL REQUIREMENTS

Apparatus:
1 x 100 mL Graduated Cylinder
2 x 250 mL Erlenmeyer Flasks
2 x Long-Stem Funnels
Whatman 12.5 cm #1 Filter Paper
1 x 25 mL Burette
1 x 1000 mL Volumetric Flask
1 x 100 mL Volumetric Flask
2 x 50 mL Beakers
4 x 150 mL Beakers
Magnetic Stir Bars
Battery Operated Magnetic Stirrer
pH Meter and Electrode

REQUIRED REAGENTS

0.1 N Nitric Acid solution
0.014 N Mercuric Nitrate solution
Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Pipet 25 mL of each filtrate into two more separate, clean 150 mL beakers. Add 75 mL of deionized water to each using the graduated cylinder.

6. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

7. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

8. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

9. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

10. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Refill the burette. Record initial burette reading. Titrate the test sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Aluminum Chloride by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x 251]

Other Aluminum Chloride compounds -

$AlCl_3$ - Concentration (mg/kg) = [a.t.v. x 251]

$AlCl_3.6\ H_2O$ - Concentration (mg/kg) = [a.t.v. x 454]

ALUMINUM CHLORIDE IN WATER AND WASTEWATER

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/L

Common Synonyms: Anhydrous Trichloroaluminum

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - sore throat, coughing, laboured breathing. Ingestion - burning sensation, stomach cramps, dizziness, diarrhea, shock, convulsions, coma. Contact - eyes- redness, pain, blurred vision; skin- redness, pain.
Toxicology: Moderately toxic, corrosive by ingestion and contact.
TLV - 5 ppm; 7 mg/cubic meter (as HCl)

EXPERIMENTAL REQUIREMENTS

Apparatus:
1 x 100 mL Graduated Cylinder
2 x 250 mL Erlenmeyer Flasks
1 x 25 mL Burette
1 x 1000 mL Volumetric Flask
1 x 100 mL Volumetric Flask
2 x 50 mL Beakers
Magnetic Stir Bars
Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

0.1 N Nitric Acid solution
0.014 N Mercuric Nitrate solution
Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

4. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

5. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

6. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

7. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial

burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Aluminum Chloride by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 12.54]

Other Aluminum Chloride compounds -

AlCl3 - Concentration (mg/L) = [a.t.v. x 12.54]

AlCl3.6 H2O - Concentration (mg/L) = [a.t.v. x 22.7]

ALUMINUM SULFATE IN SOILS

THORIN TITRIMETRIC TEST

Reference: Environment Canada, Analytical Methods Manual, 1974.

Range: 0-25000 mg/kg

Common Synonyms: Alum, Cake Alum

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - sore throat, coughing, irritation of nose and throat. Ingestion - nausea, vomiting. Contact - skin and eyes - irritated by dust.
Toxicology: Relatively non-toxic.

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 1 x 125 mL Erlenmeyer Flask fitted with a bored #6 Rubber Stopper
- 2 x Long-Stem Funnels
- Whatman 12.5 cm #1 Filter Paper
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 5 x 10 mL Disposable Pipets
- 6 x 150 mL Beakers
- 125 mL Separatory Funnel
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer
- Glass Wool
- Handpump
- pH Meter & Electrode

REQUIRED REAGENTS

- 20% Hydrochloric Acid solution
- 1% Hydrochloric Acid solution
- 1% Ammonium Hydroxide solution
- 0.00164 N Barium Chloride solution
- Ion - Exchange Resin (Dowex 50W-X8)
- Thorin indicator solution
- Ethanol

Preparation of 20% Hydrochloric Acid solution -

To a 400 mL beaker containing 200 mL of deionized water, slowly add 50 mL of concentrated hydrochloric acid.

Preparation of 1% Hydrochloric Acid -

To a 100 mL beaker containing 95 mL of deionized water, slowly add 5 mL of the 20% hydrochloric acid solution.

Preparation of 1% Ammonium Hydroxide solution -

To a 100 mL beaker containing 99 mL of deionized water, add 1 mL of concentrated ammonium hydroxide.

Preparation of 0.00164 N Barium Chloride solution -

Dissolve 0.400 g of Barium Chloride in a 500 mL beaker containing 400 mL of deionized water. Adjust the solution to pH 3.9 by adding 1% hydrochloric acid solution dropwise. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water.

Preparation of Ion-Exchange Resin -

Insert a small glass wool plug into the neck of a 125 mL separatory funnel. Add 25 g of Dowex 50W-X8 cation exchange resin.

Preparation of Thorin indicator -

Dissolve 0.2 g of Thorin in deionized water in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Prepare the ion-exchange column (as illustrated) by passing 25 mL of 20% hydrochloric acid through it. Discard the spent acid.

6. Pass 2 x 20 mL aliquots of deionized water through the ion-exchange column. Discard the spent water.

7. Pass 20 mL of the BLANK solution through the ion-exchange column. Discard the spent solution.

8. Pass a 30 mL aliquot of the BLANK solution through the ion-exchange column. Pipet 10 mL of this into a clean 150 mL beaker.

9. Repeat steps 5 through 8 using the TEST sample in place of the BLANK sample.

10. Using the graduated cylinder, add 40 mL of Ethanol to each beaker.

11. Adjust both solutions to pH 3.8-4.0 using 1% hydrochloric acid or 1% ammonium hydroxide as required.

12. Add 2-3 drops of thorin indicator to each beaker. The solution should now be orange in colour.

13. Fill the 0-25 mL burette with the barium chloride solution. Record the initial burette reading.

14. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

15. Titrate the BLANK solution by slowly adding the barium chloride until the first permanent pink colour appears. Record the final volume.

16. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Aluminum Sulfate by solving the following equation:

$$\text{Concentration (mg/kg)} = [\ (\text{adjusted titrant volume}) \times 188\ ]$$

Other Aluminum Sulfate compounds -

$Al_2(SO_4)_3$ - Concentration (mg/kg) = [a.t.v. x 188]

$Al_2(SO_4)_3.18\ H_2O$ - Concentration (mg/kg) = [a.t.v. x 365]

AMMONIUM CHLORIDE IN SOILS

MERCURIC NITRATE TITRIMETRIC METHOD

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/kg

Common Synonyms: Sal Ammoniac, Ammonium Muriate.

HAZARDOUS DATA

Human Health
Symptoms: Contact - eyes and skin - irritating. Inhalation-irritates nose, throat, respiratory passages. Ingestion - nausea and vomiting.
Toxicology: Moderately toxic upon inhalation and ingestion.
TLV - 10 mg/cubic meter (fume)
Short-term inhalation limits - 20 mg/cubic meter (fume - 15 min).

EXPERIMENTAL REQUIREMENTS

Apparatus:
1 x 100 mL Graduated Cylinder
2 x 250 mL Erlenmeyer Flasks
2 x Long-Stem Funnels
Whatman 12.5 cm #1 Filter Paper
1 x 25 mL Burette
1 x 1000 mL Volumetric Flask
1 x 100 mL Volumetric Flask
2 x 50 mL Beakers
4 x 150 mL Beakers
Magnetic Stir Bars
Battery Operated Magnetic Stirrer
pH Meter and Electrode

REQUIRED REAGENTS

0.1 N Nitric Acid solution
0.014 N Mercuric Nitrate solution
Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Pipet 25 mL of each filtrate into two more separate, clean 150 mL beakers. Add 75 mL of deionized water to each using the graduated cylinder.

6. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

7. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

8. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

9. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

10. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Refill the burette. Record initial titrant volume. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Ammonium Chloride by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x 302]

AMMONIUM CHLORIDE IN WATER AND WASTEWATER

MERCURIC NITRATE TITRIMETRIC METHOD

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/L

Common Synonyms: Sal Ammoniac, Ammonium Muriate.

HAZARDOUS DATA

Human Health
Symptoms: Contact - eyes and skin - irritating. Inhalation-irritates nose, throat, respiratory passages. Ingestion - nausea and vomiting.
Toxicology: Moderately toxic upon inhalation and ingestion.
TLV - 10 mg/cubic meter (fume)
Short-term inhalation limits - 20 mg/cubic meter (fume - 15 min).

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 1 x 100 mL Volumetric Flask
- 2 x 50 mL Beakers
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

- 0.1 N Nitric Acid solution
- 0.014 N Mercuric Nitrate solution
- Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of

deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

4. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

5. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

6. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

7. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

8. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Ammonium Chloride by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 15.1]

AMMONIUM NITRATE IN SOILS

CADMIUM REDUCTION COLOURIMETRIC METHOD

Reference: Hach Manual (Nitrogen, Nitrate High Range)

Range: 100 - 4000 mg/kg

Common Synonyms: Nitric Acid,Ammonium Salt, German Saltpeter, Norway Saltpeter, Nitram.

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin and eyes- irritation of eyes and mucous membranes. Inhalation - sore throat, coughing, shortness of breath. Ingestion - dizziness, cramps, and vomiting.
Toxicology: Moderately toxic by contact and ingestion.

Environment: Harmful to aquatic life in high concentrations.
Aquatic Toxicity Rating - 10 to 100/96h/TLm/freshwater
LD50 (oral, rat) - 4500 mg/kg

Detonates under certain conditions.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Beakers
2 x Magnetic Stirring Bars
Battery-Operated Magnetic Stirrer
2 x 250 mL Erlenmeyer Flasks
2 x Funnels
Whatman #1 Filter Paper

REQUIRED REAGENTS

Nitraver V Reagent (included with Hach kit)

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil

from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Put a magnetic stirring bar into each beaker. Add the contents of one pouch of NitraVer 5 Nitrate Reagent Powder Pillow to each of the beakers. Put the Blank beaker on the magnetic stirrer and turn on for one minute.

4. Replace the Blank beaker with the TEST flask and stir the TEST beaker for one minute also.

5. Adjust the Wavelength Dial of the spectrophotometer to read 500 nm.

6. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

7. Replace the Zero Scale card with the AMMONIUM NITRATE IN SOIL card.

8. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

9. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

10. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Ammonium Nitrate concentration from the card and record this result.

AMMONIUM NITRATE IN WATER AND WASTEWATER

CADMIUM REDUCTION COLOURIMETRIC METHOD

Reference: Hach Manual (Nitrogen, Nitrate High Range)

Range: 0 - 80 mg/L

Common Synonyms: Nitric Acid,Ammonium Salt, German Saltpeter, Norway Saltpeter, Nitram.

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin and eyes- irritation of eyes and mucous membranes. Inhalation - sore throat, coughing, shortness of breath. Ingestion - dizziness, cramps, and vomiting.
Toxicology: Moderately toxic by contact and ingestion.

Environment: Harmful to aquatic life in high concentrations.
Aquatic Toxicity Rating - 10 to 100/96h/TLm/freshwater
LD50 (oral, rat) - 4500 mg/kg

Detonates under certain conditions.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Beakers
2 x Magnetic Stirring Bars
Battery-Operated Magnetic Stirrer

REQUIRED REAGENTS

Nitraver V Reagent (included with Hach kit)

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer beaker. Label the beaker BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer beaker. Label the beaker TEST.

3. Put a magnetic stirring bar into each beaker. Add the contents of one pouch of NitraVer 5 Nitrate Reagent Powder Pillow to each of the beakers. Put the Blank beaker on the magnetic stirrer and turn on for one minute.

4. Replace the Blank beaker with the TEST flask and stir the TEST beaker for one minute also.

5. Adjust the Wavelength Dial of the spectrophotometer to read 500 nm.

6. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

7. Replace the Zero Scale card with the AMMONIUM NITRATE IN WATER card.

8. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

9. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

10. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Ammonium Nitrate concentration from the card and record this result.

AMMONIUM PHOSPHATES IN WATER AND WASTEWATER

AMINO ACID COLORIMETRIC TEST

Reference: APHA Standard Methods, 12 Ed.,1965.

Range: 0-25 mg/L

Common Synonyms: Ammonium Phosphate Monobasic - Ammonium Acid Phosphate, Ammonium Biphosphate, Ammonium Dihydrogen Phosphate, Ammonium Phosphate Primary.

Ammonium Phosphate Dibasic - Ammonium Phosphate Secondary, Ammonium Hydrogen Phosphate.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - monobasic form causes irritation of mucous membranes; dibasic form- ammonia vapours in closed area can cause lung irritation and asphyxia. Contact - skin and eye irritation.
Toxicology: Low order of toxicity by all routes.

Environment:
Aquatic Toxicity Rating - 100 to 1000 ppm/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 25 mL Graduated Cylinders
5 x 1 mL Disposable Pipets
Timing Device
2 x 250 mL Erlenmeyer Flasks

REQUIRED REAGENTS

Molybdate Reagent
Amino Acid pillows
*Note: all required reagents supplied by Hach Company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Pipet 1.0 mL of Molybdate Reagent into each flask.

4. Add 1 Amino Acid pillow to each flask and mix thoroughly. Wait 10 minutes for colour stabilization.

5. Adjust the Wavelength Dial of the spectrophotometer to read 530 nm.

6. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

7. Replace the Zero Scale card with the Ammonium Phosphate card.

8. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

9. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

10. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Ammonium Phosphate concentration from the card and record this result.

AMMONIUM SULFATE IN SOILS

THORIN TITRIMETRIC TEST

Reference: Environment Canada, Analytical Methods Manual, 1974.

Range: 0-25000 mg/kg

Common Synonyms: Diammonium Sulfate

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation from dust. Ingestion- abdominal pain and nausea.
Toxicology: Relatively non-toxic.

EXPERIMENTAL REQUIREMENTS

Apparatus: 1 x 100 mL Graduated Cylinder
2 x 250 mL Erlenmeyer Flasks
1 x 125 mL Erlenmeyer Flask fitted with a bored #6 Rubber Stopper
2 x Long-Stem Funnels
Whatman 12.5 cm #1 Filter Paper
1 x 25 mL Burette
1 x 1000 mL Volumetric Flask
5 x 10 mL Disposable Pipets
6 x 150 mL Beakers
125 mL Separatory Funnel
Magnetic Stir Bars
Battery Operated Magnetic Stirrer
Glass Wool
Handpump
pH Meter & Electrode

REQUIRED REAGENTS

20% Hydrochloric Acid solution
1% Hydrochloric Acid solution
1% Ammonium Hydroxide solution
0.00164 N Barium Chloride solution
Ion - Exchange Resin (Dowex 50W-X8)
Thorin indicator solution
Ethanol

Preparation of 20% Hydrochloric Acid solution -

To a 400 mL beaker containing 200 mL of deionized water, slowly add 50 mL of concentrated hydrochloric acid.

Preparation of 1% Hydrochloric Acid -

To a 100 mL beaker containing 95 mL of deionized water, slowly add 5 mL of the 20% hydrochloric acid solution.

Preparation of 1% Ammonium Hydroxide solution -

To a 100 mL beaker containing 99 mL of deionized water, add 1 mL of concentrated ammonium hydroxide.

Preparation of 0.00164 N Barium Chloride solution -

Dissolve 0.400 g of Barium Chloride in a 500 mL beaker containing 400 mL of deionized water. Adjust the solution to pH 3.9 by adding 1% hydrochloric acid solution dropwise. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water.

Preparation of Ion-Exchange Resin -

Insert a small glass wool plug into the neck of a 125 mL separatory funnel. Add 25 g of Dowex 50W-X8 cation exchange resin.

Preparation of Thorin indicator -

Dissolve 0.2 g of Thorin in deionized water in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Prepare the ion-exchange column (as illustrated) by passing 25 mL of 20% hydrochloric acid through it. Discard the spent acid.

6. Pass 2 x 20 mL aliquots of deionized water through the ion-exchange column. Discard the spent water.

7. Pass 20 mL of the BLANK solution through the ion-exchange column. Discard the spent solution.

8. Pass a 30 mL aliquot of the BLANK solution through the ion-exchange column. Pipet 10 mL of this into a clean 150 mL beaker.

9. Repeat steps 5 through 8 using the TEST sample in place of the BLANK sample.

10. Using the graduated cylinder, add 40 mL of Ethanol to each beaker.

11. Adjust both solutions to pH 3.8-4.0 using 1% hydrochloric acid or 1% ammonium hydroxide as required.

12. Add 2-3 drops of thorin indicator to each beaker. The solution should now be orange in colour.

13. Fill the 0-25 mL burette with the barium chloride solution. Record the initial burette reading.

14. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

15. Titrate the BLANK solution by slowly adding the barium chloride until the first permanent pink colour appears. Record the final volume.

16. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Ammonium Sulfate by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x 220]

BENZENE IN SOILS

TETRACYANOETHYLENE COLORIMETRIC TEST

Reference: Schinck, G.H., Ozolins, M., Complexometric Determination of Aromatic Hydrocarbons with Tetracyanoethylene, Anal. Chem., 33, 11, 1961.

Range: 0-15000 mg/kg

Common Synonyms: Benzol, Coal Naphtha, Benzin, Phenylhydride.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation, headache, dizziness, dullness, nausea, respiratory paralysis, unconsciousness. Contact - skin- irritation, prolonged exposure same symptoms as inhalation. Ingestion - sore throat, nausea, vomiting, headache, unconsciousness.
Toxicology: Toxic by all routes.
TLV - Inhalation: 10 ppm; 30 mg/cubic meter

Environment:
Aquatic Toxicity Rating - 10 to 100 ppm/96h/TLm/freshwater;
BOD: 1.2 lb/lb, 10 days.

EXPERIMENTAL REQUIREMENTS

Apparatus:
- Hach Spectrophotometer and 2 Cuvettes
- 2 x 250 mL Erlenmeyer Flasks
- 2 x 150 mL Beakers
- 2 x 50 mL Stoppered Graduated Mixing Cylinders
- 2 x 25 mL Graduated Cylinders
- 1 x 200 mL Volumetric Flask
- 5 x 5 mL Disposable Pipets
- 1 x 100 mL Beaker
- Timing Device
- Whatman #1 Filter Paper
- 2 x Long-stemmed Funnels
- Balance

REQUIRED REAGENTS

0.1 M Tetracyanoethylene Solution
Chloroform

Preparation of 0.1 M Tetracyanoethylene solution -

Dissolve 2.562 g of tetracyanoethylene in 10 mL of acetone in a 100 mL beaker. Transfer the solution to a 200 mL volumetric flask with methylene chloride. Dilute to the 200 mL mark with methylene chloride. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of Chloroform to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Transfer 20 mL of each filtrate to separate 50 mL mixing cylinders.

6. Pipet 5 mL of the tetracyanoethylene solution into each mixing cylinder, stopper, and mix well. Wait 2-3 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the spectrophotometer to read 440 nm.

8. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

9. Replace the Zero Scale card with the BENZENE in SOIL card.

10. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

11. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

12. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Benzene in Soil concentration from the card and record this result.

BENZENE IN WATER AND WASTEWATER

TETRACYANOETHYLENE COLORIMETRIC TEST

Reference: Schinck, G.H., Ozolins, M., Complexometric Determination of Aromatic Hydrocarbons with Tetracyanoethylene, Anal. Chem., 33, 11, 1961.

Range: 0-15000 mg/L

Common Synonyms: Benzol, Coal Naphtha, Benzin, Phenylhydride.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation, headache, dizziness, dullness, nausea, respiratory paralysis, unconsciousness. Contact - skin- irritation, prolonged exposure same symptoms as inhalation. Ingestion - sore throat, nausea, vomiting, headache, unconsciousness.
Toxicology: Toxic by all routes.
TLV - Inhalation: 10 ppm; 30 mg/cubic meter

Environment:
Aquatic Toxicity Rating - 10 to 100 ppm/96h/TLm/freshwater;
BOD: 1.2 lb/lb, 10 days.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 150 mL Beakers
2 x 125 mL Separatory Funnels
2 x 50 mL Stoppered Graduated Mixing Cylinders
2 x 25 mL Graduated Cylinders
1 x 200 mL Volumetric Flask
5 x 5 mL Disposable Pipets
1 x 100 mL Beaker
Timing Device
Balance

REQUIRED REAGENTS

0.1 M Tetracyanoethylene Solution
Chloroform

Preparation of 0.1 M Tetracyanoethylene solution -

Dissolve 2.562 g of tetracyanoethylene in 10 mL of acetone in a 100 mL beaker. Transfer the solution to a 200 mL volumetric flask with methylene chloride. Dilute to the 200 mL mark with methylene chloride. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 25 mL water sample in a graduated cylinder. Pour the sample into a clean 125 mL separatory funnel. Label the funnel BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 25 mL mark. Pour the water into a second clean 125 mL separatory funnel. Label the funnel TEST.

3. Pipet 25 mL of chloroform into each of the 125 mL separatory funnels, stopper them, and shake well. Invert the separatory funnel periodically and open the stopcock to vent any buildup of gases.

4. Allow the phases to separate (usually 2-3 minutes).

5. Drain from each funnel 25 mL of the bottom (chloroform) layer into separate graduated mixing cylinders.

6. Pipet 5 mL of the tetracyanoethylene solution into each mixing cylinder, stopper, and mix well. Wait 2-3 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

8. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

9. Replace the Zero Scale card with the BENZENE in WATER card.

10. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

11. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

12. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Benzene in Water concentration from the card and record this result.

BENZOIC ACID IN WATER

SODIUM HYDROXIDE TITRATION METHOD

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0 - 10000 mg/L

Common Synonyms: Benzenecarboxylic Acid, Carboxybenzene, Phenylformic Acid, Phenylcarboxylic Acid.

HAZARDOUS DATA

Human Health
Symptoms: Contact - irritation of skin; corrosive and irritating to eyes. Inhalation - irritation of respiratory tract, nose and throat. Ingestion - nausea, gastrointestinal problems.
Toxicology: Moderately toxic by all routes.

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 50 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
1 x 25 mL Burette

REQUIRED REAGENTS

Phenolphthalein indicator solution
0.02N Sodium Hydroxide solution
*Note: All required reagents supplied by Hach company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Fill the 0 - 25 mL burette with 0.02N Sodium Hydroxide

solution and record the initial burette reading.

4. Add 6 drops of phenolphthalien solution to each flask.

5. Titrate the BLANK water sample against the sodium hydroxide solution; add slowly, with continuous swirling until a pink colour appears for more than 30 seconds.

6. Record the final burette reading.

7. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Benzoic Acid by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 48]

BORIC ACID IN SOIL

HYDROLYSIS AND SODIUM HYDROXIDE TITRATION

Reference: Smith, D.M., Bryant, W.M.D., J. Am. Chem. Soc., 58, 2452, 1936.

Range: 500 - 5000 mg/kg

Common Synonyms: Boracic acid, Orthoboric acid, Borofax.

HAZARDOUS DATA

Human Health
Symptoms: Ingestion - nausea, vomiting, diarrhea, abdominal cramps, erythematous lesions on skin and mucous membranes, circulatory collapse, tachycardia, cyanosis, delirium, convulsions, coma. Contact - toxic by skin absorption, rash, blistering.
Toxicology: Highly toxic by oral route, moderate by others.

Environment:
Aquatic Toxicity Rating - 10 to 100/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 125 Erlenmeyer flasks
- 2 x funnel
- filter paper
- 2 x 150 mL beaker
- 50 mL graduated cylinder
- 2 x magnetic stirring bars
- magnetic stirrer
- 25 mL buret

REQUIRED REAGENTS

- 10% Pyridine solution
- 0.02N Sodium Hydroxide
- Thymol blue indicator solution

Preparation of 10% pyridine solution -

Combine 135 mL of deionized water with 15 mL of pyridine in a 500 mL bottle with a screw-top lid.

Preparation of Thymol blue indicator solution -

Dissolve 0.4 grams of Thymol blue in 100 mL of deionized water and store in a 150 mL plastic bottle with lid.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of 10% pyridine solution to each flask. Mix well for 5 minutes.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Put a magnetic stirring bar in each beaker. Add 0.5 mL of Thymol blue indicator to each sample.

6. Fill the buret with 0.02N Sodium Hydroxide solution. Record the initial buret reading. Place the BLANK sample on the magnetic stirrer and turn stirrer on to low speed. Titrate the BLANK sample with Sodium Hydroxide until the solution changes colour from yellow to blue-green. Record the final buret reading.

7. Refill the buret. Record initial buret reading. Titrate the TEST sample as above. Record final buret reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Boric acid by solving the following equation:

$$\text{Concentration (mg/kg)} = [\ (\text{adjusted titrant volume}) \times 480\ ]$$

1-BUTANOL IN SOIL

CERIC AMMONIUM NITRATE METHOD

Reference: Reid, V.W., Truelove, R.K., The Colorimetric Determination of Alcohols, The Analyst, Vol. 77, 325, 1952.

Range: 2000 - 40000 mg/kg

Common Synonyms: n-Butyl Alcohol, Butyl alcohol, propyl carbinol, Butyl Hydroxide, Butyric Alcohol, Hydroxybutane.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - headache, dizziness, nausea, narcosis. Ingestion - abdominal pains, nausea. Contact - absorbed readily through skin and eyes, contact dermatitis.
Toxicology: Moderately toxic by all routes.

Environment:
Aquatic Toxicity Rating - >1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer flasks
2 x funnels
filter paper
2 x 150 mL beakers
50 mL graduated cylinder
Hach spectrophotometer
2 x cuvettes
graduated 5 mL pipet

REQUIRED REAGENTS

Ceric Ammonium Nitrate Solution

Preparation of Ceric ammonium nitrate solution -

Dissolve 43.27 grams of Ceric ammonium nitrate powder in 100 mL of deionized water in a 150 mL beaker. When fully dissolved, transfer to a 100 mL reagent bottle with screwtop lid.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Add 2 mL of Ceric ammonium nitrate solution to each beaker.

6. Adjust the Wavelength Dial of the spectrophotometer to read 460 nm.

7. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

8. Replace the Zero Scale card with the 1-BUTANOL in SOIL card.

9. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

10. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

11. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the 1-Butanol concentration from the card and record this result.

1-BUTANOL IN WATER

CERIC AMMONIUM NITRATE METHOD

Reference: Reid, V.W., Truelove, R.K., The Colorimetric Determination of Alcohols, The Analyst, Vol. 77, 325, 1952.

Range: 200 - 8000 mg/L

Common Synonyms: n-Butyl Alcohol, Butyl alcohol, propyl carbinol, Butyl Hydroxide, Butyric Alcohol, Hydroxybutane.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - headache, dizziness, nausea, narcosis. Ingestion - abdominal pains, nausea. Contact - absorbed readily through skin and eyes, contact dermatitis.
Toxicology: Moderately toxic by all routes.

Environment:
Aquatic Toxicity Rating - >1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 150 mL beakers
50 mL graduated cylinder
Hach spectrophotometer
2 x cuvettes
graduated 5 mL pipet

REQUIRED REAGENTS

Ceric Ammonium Nitrate Solution

Preparation of Ceric ammonium nitrate solution -

Dissolve 43.27 grams of Ceric ammonium nitrate powder in 100 mL of deionized water in a 150 mL beaker. When fully dissolved, transfer to a 100 mL reagent bottle with screwtop lid.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean

150 mL beaker. Label the beaker BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 150 mL beaker. Label the beaker TEST.

3. Add 2 mL of Ceric ammonium nitrate solution to each beaker.

4. Adjust the Wavelength Dial of the spectrophotometer to read 450 nm.

5. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

6. Replace the Zero Scale card with the 1-BUTANOL in WATER card.

7. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

8. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

9. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the 1-Butanol concentration from the card and record this result.

iso-BUTYRALDEHYDE IN SOIL

2,4-DINITROPHENYLHYDRAZINE METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Permagon Press, 1963.

Range: 0 - 150 mg/kg

Common Synonyms: iso-Butanol, iso-Butyric Aldehyde.

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin- burning sensation and blistering; eyes- watering, burning sensation. Inhalation - irritation of mucous membranes, difficulty breathing, headache, cyanosis, nausea, muscular weakness. Ingestion - burning sensation, difficulty breathing, nausea, vomiting, convulsions.
Toxicology: Moderately toxic by all dermal routes.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
2 x 100 mL Mixing Cylinders
5 x 10 mL Disposable Pipets
2 x 150 mL Beakers
1 x 100 mL Volumetric Flask
Timing Device
Whatman #1 Filter Paper
2 x Long Stemmed Funnels
Balance

REQUIRED REAGENTS

Pyridine Stabilizer
33% Potassium Hydroxide Solution
2,4-Dinitrophenylhydrazine Solution

Preparation of Pyridine Stabilizer

Add 100 mL of deionized water to 400 mL of pyridine. Mix thouroughly. Store in a brown glass bottle.

Preparation of 33% Potassium Hydroxide Solution

Dissolve 33.0 g of potassium hydroxide in a 150 mL beaker containing 100 mL of methanol.

Preparation of 2,4-Dinitrophenylhydrazine solution

Dissolve 0.100 g of 2,4-Dinitrophenylhydrazine in a 100 mL beaker containing 50 mL of methanol and 4 mL of concentrated hydrochloric acid. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Transfer 50 mL of each filtrate into separate mixing cylinders.

6. Pipet 1.0 mL of the 2,4-Dinitrophenylhydrazine solution into each mixing cylinder. Stopper and shake gently. Wait exactly 20 minutes.

7. Pipet 1.0 mL of the 33% potassium hydroxide solution into each mixing cylinder. Then add 5 mL of pyridine stabilizer to each and stopper. Mix well and wait 2 minutes for colour stabilization.

8. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

9. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

10. Replace the Zero Scale card with the iso-BUTYRALDEHYDE in SOIL card.

11. Fill one cuvette to the 25 mL mark from the cylinder labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

12. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

13. Fill the other cuvette to the 25 mL mark from the cylinder labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the isobutyraldehyde concentration from the card and record this result.

iso-BUTYRALDEHYDE IN WATER AND WASTEWATER

2,4-DINITROPHENYLHYDRAZINE METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Permagon Press, 1963.

Range: 0 - 40 mg/L

Common Synonyms: iso-Butanol, iso-Butyric Aldehyde.

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin- burning sensation and blistering; eyes- watering, burning sensation. Inhalation - irritation of mucous membranes, difficulty breathing, headache, cyanosis, nausea, muscular weakness. Ingestion - burning sensation, difficulty breathing, nausea, vomiting, convulsions.
Toxicology: Moderately toxic by all dermal routes.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
5 x 10 mL Disposable Pipets
2 x 150 mL Beakers
1 x 100 mL Volumetric Flask
Timing Device

REQUIRED REAGENTS

Pyridine Stabilizer
33% Potassium Hydroxide Solution
2,4-Dinitrophenylhydrazine Solution

Preparation of Pyridine Stabilizer

Add 100 mL of deionized water to 400 mL of pyridine. Mix thouroughly. Store in a brown glass bottle.

Preparation of 33% Potassium Hydroxide Solution

Dissolve 33.0 g of potassium hydroxide in a 150 mL beaker containing 100 mL of methanol.

Preparation of 2,4-Dinitrophenylhydrazine solution

Dissolve 0.100 g of 2,4-Dinitrophenylhydrazine in a 100 mL beaker containing 50 mL of methanol and 4 mL of concentrated hydrochloric acid. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. From an uncontaminated source in the area obtain a 100 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 100 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Pipet 2.0 mL of the 2,4-Dinitrophenylhydrazine solution into each flask. Stopper and shake gently. Wait exactly 20 minutes.

7. Pipet 2.0 mL of the 33% potassium hydroxide solution into each flask. Then add 10 mL of pyridine stabilizer to each and mix well and wait 2-3 minutes for colour stabilization.

8. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

9. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

10. Replace the Zero Scale card with the iso-BUTYRALDEHYDE in WATER card.

11. Fill one cuvette to the 25 mL mark from the flask labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

12. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

13. Fill the other cuvette to the 25 mL mark from the flask labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the iso-butyraldehyde concentration from the card and record this result.

n-BUTYRALDEHYDE IN SOIL

2,4-DINITROPHENYLHYDRAZINE METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Permagon Press, 1963.

Range: 0 - 200 mg/kg

Common Synonyms: Butyric Aldehyde, n-Butanal.

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin- burning sensation and blistering; eyes- watering, burning sensation. Inhalation - irritation of mucous membranes, difficulty breathing, headache, cyanosis, nausea, muscular weakness. Ingestion - burning sensation, difficulty breathing, nausea, vomiting, convulsions.
Toxicology: Moderately toxic by all dermal routes.

Environmental
Aquatic toxicity rating - 1 to 10 ppm/96h/TLm/freshwater
BOD:106%, 5 days.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
2 x 100 mL Mixing Cylinders
5 x 10 mL Disposable Pipets
2 x 150 mL Beakers
1 x 100 mL Volumetric Flask
Timing Device
Whatman #1 Filter Paper
2 x Long Stemmed Funnels
Balance

REQUIRED REAGENTS

Pyridine Stabilizer
33% Potassium Hydroxide Solution
2,4-Dinitrophenylhydrazine Solution

Preparation of Pyridine Stabilizer

Add 100 mL of deionized water to 400 mL of pyridine. Mix thoroughly. Store in a brown glass bottle.

Preparation of 33% Potassium Hydroxide Solution

Dissolve 33.0 g of potassium hydroxide in a 150 mL beaker containing 100 mL of methanol.

Preparation of 2,4-Dinitrophenylhydrazine solution

Dissolve 0.100 g of 2,4-Dinitrophenylhydrazine in a 100 mL beaker containing 50 mL of methanol and 4 mL of concentrated hydrochloric acid. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Transfer 50 mL of each filtrate into separate mixing cylinders.

6. Pipet 1.0 mL of the 2,4-Dinitrophenylhydrazine solution into each mixing cylinder. Stopper and shake gently. Wait exactly 20 minutes.

7. Pipet 1.0 mL of the 33% potassium hydroxide solution into each mixing cylinder. Then add 5 mL of pyridine stabilizer to each and stopper. Mix well and wait 2 minutes for colour stabilization.

8. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

9. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

10. Replace the Zero Scale card with the n-BUTYRALDEHYDE in SOIL card.

11. Fill one cuvette to the 25 mL mark from the cylinder labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

12. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

13. Fill the other cuvette to the 25 mL mark from the cylinder labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the n-butyraldehyde concentration from the card and record this result.

n-BUTYRALDEHYDE IN WATER AND WASTEWATER

2,4-DINITROPHENYLHYDRAZINE METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Permagon Press, 1963.

Range: 0 - 40 mg/L

Common Synonyms: Butyric Aldehyde, n-Butanal.

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin- burning sensation and blistering; eyes- watering, burning sensation. Inhalation - irritation of mucous membranes, difficulty breathing, headache, cyanosis, nausea, muscular weakness. Ingestion - burning sensation, difficulty breathing, nausea, vomiting, convulsions.
Toxicology: Moderately toxic by all dermal routes.

Environmental
Aquatic toxicity rating - 1 to 10 ppm/96h/TLm/freshwater
BOD:106%, 5 days.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
5 x 10 mL Disposable Pipets
2 x 150 mL Beakers
1 x 100 mL Volumetric Flask
Timing Device

REQUIRED REAGENTS

Pyridine Stabilizer
33% Potassium Hydroxide Solution
2,4-Dinitrophenylhydrazine Solution

Preparation of Pyridine Stabilizer

Add 100 mL of deionized water to 400 mL of pyridine. Mix thoroughly. Store in a brown glass bottle.

Preparation of 33% Potassium Hydroxide Solution

Dissolve 33.0 g of potassium hydroxide in a 150 mL beaker containing 100 mL of methanol.

Preparation of 2,4-Dinitrophenylhydrazine solution

Dissolve 0.100 g of 2,4-Dinitrophenylhydrazine in a 100 mL beaker containing 50 mL of methanol and 4 mL of concentrated hydrochloric acid. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. From an uncontaminated source in the area obtain a 100 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 100 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Pipet 2.0 mL of the 2,4-Dinitrophenylhydrazine solution into each flask. Stopper and shake gently. Wait exactly 20 minutes.

4. Pipet 2.0 mL of the 33% potassium hydroxide solution into each flask. Then add 10 mL of pyridine stabilizer to each and mix well and wait 2-3 minutes for colour stabilization.

5. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

6. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

7. Replace the Zero Scale card with the n-BUTYRALDEHYDE in WATER card.

8. Fill one cuvette to the 25 mL mark from the flask labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

9. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

10. Fill the other cuvette to the 25 mL mark from the flask labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the n-butyraldehyde concentration from the card and record this result.

CALCIUM CHLORIDE IN SOILS

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/kg

Common Synonyms: Anhydrous Calcium Chloride

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of nose and throat. Ingestion - irritation of mouth and stomach, nausea and vomiting. Contact - eyes- irritation from dust, possible transient corneal injury; skin- dry solid causes mild irritation; strong solutions cause marked irritation, even superficial burns.

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 2 x Long-Stem Funnels
- Whatman 12.5 cm #1 Filter Paper
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 1 x 100 mL Volumetric Flask
- 2 x 50 mL Beakers
- 4 x 150 mL Beakers
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer
- pH Meter and Electrode

REQUIRED REAGENTS

- 0.1 N Nitric Acid solution
- 0.014 N Mercuric Nitrate solution
- Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and

label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Pipet 25 mL of each filtrate into two more separate, clean 150 mL beakers. Add 75 mL of deionized water to each using the graduated cylinder.

6. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

7. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

8. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

9. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

10. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Calcium Chloride by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x 313]

Other Calcium Chloride compounds -

$CaCl_2$ - Concentration (mg/kg) = [a.t.v. x 313]

$CaCl_2 \cdot H_2O$ - Concentration (mg/kg) = [a.t.v. x 364]

$CaCl_2 \cdot 2\,H_2O$ - Concentration (mg/kg) = [a.t.v. x 415]

$CaCl_2 \cdot 6\,H_2O$ - Concentration (mg/kg) = [a.t.v. x 618]

CALCIUM CHLORIDE IN WATER AND WASTEWATER

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/L

Common Synonyms: Anhydrous Calcium Chloride

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of nose and throat. Ingestion - irritation of mouth and stomach, nausea and vomiting. Contact - eyes- irritation from dust, possible transient corneal injury; skin- dry solid causes mild irritation; strong solutions cause marked irritation, even superficial burns.

EXPERIMENTAL REQUIREMENTS

Apparatus:
1 x 100 mL Graduated Cylinder
2 x 250 mL Erlenmeyer Flasks
1 x 25 mL Burette
1 x 1000 mL Volumetric Flask
1 x 100 mL Volumetric Flask
2 x 50 mL Beakers
Magnetic Stir Bars
Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

0.1 N Nitric Acid solution
0.014 N Mercuric Nitrate solution
Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

4. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

5. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

6. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

7. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Repeat the titration with the TEST solution, making sure to record both initial and final burette readings.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and

TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Calcium Chloride by solving the following equation:

$$\text{Concentration (mg/L)} = \frac{[\ (\text{adjusted titrant volume}) \times 15.65\ ]}{}$$

Other Calcium Chloride compounds -

$CaCl_2$ - Concentration (mg/L) = [a.t.v. x 15.65]

$CaCl_2 \cdot H_2O$ - Concentration (mg/L) = [a.t.v. x 18.2]

$CaCl_2 \cdot 2\ H_2O$ - Concentration (mg/L) = [a.t.v. x 20.73]

$CaCl_2 \cdot 6\ H_2O$ - Concentration (mg/L) = [a.t.v. x 30.9]

CALCIUM HYDROXIDE IN WATER AND WASTEWATER

SULFURIC ACID TITRATION

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0-10000 mg/L

Common Synonyms: Lime, Calcium Hydrate, Hydrated Lime, Lime Hydrate, Slaked Lime, Caustic Lime, Agricultural Lime.

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin- burning sensation, inflammation; eyes- pain and watering. Inhalation - irritation of respiratory tract, difficulty breathing, coughing, sneezing. Ingestion- burning sensation, pain, stomach cramps.
Toxicology: TLV - (inhalation) 5 mg/cubic meter

Environment:
Aquatic toxicity rating - 10 to 1000 ppm/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer Flasks
2 x 50 mL Graduated Cylinders
1 x 25 mL Burette
Magnetic Stir Bars
Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

0.02 N Sulfuric Acid
Phenolphthalein indicator solution
Bromocresol Green - Methyl Red indicator solution
*Note: All required reagents are supplied by the Hach Company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Fill the burette with 0.02 N Sulfuric Acid and record the initial burette reading.

4. Put a magnetic stir bar in the BLANK flask, put the flask on the stirrer and turn ON. Add 6 drops of the phenolphthalein indicator to the BLANK solution and mix. A pink colour should develop; if the solution remains colourless, proceed to step 6.

5. Titrate the BLANK sample by slowly adding the 0.02 N sulfuric acid from the burette. Stop the titration when the water sample becomes colourless.

6. Add 6 drops of the bromocresol green - methyl red indicator to the BLANK solution. A blue-green colour will appear upon addition of the indicator.

7. Continue (or start) titrating with 0.02 N sulfuric acid until the solution turns a pink-orange colour. Record the final burette reading.

8. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Calcium Hydroxide by solving the following equation:

$$\text{Concentration (mg/L)} = [\ (\text{adjusted titrant volume}) \times 17\ ]$$

CALCIUM OXIDE IN WATER AND WASTEWATER

SULFURIC ACID TITRATION

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0-10000 mg/L

Common Synonyms: Burnt Lime, Calx, Quick Lime, Lime, Unslaked Lime, Pebble Lime.

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin- pain, ulceration, shock; eyes- pain, watering, burns, ulceration of eyes, perforation, blindness. Inhalation - burning sensation in nose and throat, difficulty breathing, coughing, chemical bronchitis. Ingestion - burning sensation in mouth, throat, stomach pain and cramps, vomiting, diarrhea, unconsciousness.
Toxicology: Toxic by all routes.
TLV - 2 mg/cubic meter (dust).

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer Flasks
2 x 50 mL Graduated Cylinders
1 x 25 mL Burette
Magnetic Stir Bars
Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

0.02 N Sulfuric Acid
Phenolphthalein indicator solution
Bromocresol Green - Methyl Red indicator solution
*Note: All required reagents are supplied by the Hach Company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Fill the burette with 0.02 N Sulfuric Acid and record the initial burette reading.

4. Put a magnetic stir bar in the BLANK flask, place it on the magnetic stirrer and turn ON. Add 6 drops of the phenolphthalein indicator to the BLANK solution and mix. A pink colour should develop; if the solution remains colourless, proceed to step 6.

5. Titrate the BLANK sample by slowly adding the 0.02 N sulfuric acid from the burette. Stop the titration when the water sample becomes colourless.

6. Add 6 drops of the bromocresol green - methyl red indicator to the BLANK solution. A blue-green colour will appear upon addition of the indicator.

7. Continue (or start) titrating with 0.02 N sulfuric acid until the solution turns a pink-orange colour. Record the final burette reading.

8. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Calcium Oxide by solving the following equation:

$$\text{Concentration (mg/L)} = [\ (\text{adjusted titrant volume}) \times 15.2\ ]$$

CALCIUM PHOSPHATE IN WATER AND WASTEWATER

AMINO ACID COLORIMETRIC TEST

Reference: APHA Standard Methods, 12 Ed.,1965.

Range: 0-25 mg/L

Common Synonyms: Monobasic Calcium Phosphate, Calcium Biphosphate, Acid Calcium Phosphate, Calcium Phosphate Primary.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of upper respiratory tract.
Ingestion - may cause nausea, vomiting, stomach cramps, diarrhea.
Contact - skin and eye irritation and redness.
Toxicology: Relatively non-toxic by all routes.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 25 mL Graduated Cylinders
5 x 1 mL Disposable Pipets
Timing Device
2 x 250 mL Erlenmeyer Flasks

REQUIRED REAGENTS

Molybdate Reagent
Amino Acid pillows
*Note: all required reagents supplied by Hach Company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Pipet 1.0 mL of Molybdate Reagent into each flask.

4. Add 1 Amino Acid pillow to each flask and mix thoroughly. Wait 10 minutes for colour stabilization.

5. Adjust the Wavelength Dial of the spectrophotometer to read 530 nm.

6. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

7. Replace the Zero Scale card with the Calcium Phosphate card.

8. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

9. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

10. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Calcium Phosphate concentration from the card and record this result.

CUMENE IN SOILS

TETRACYANOETHYLENE COLORIMETRIC TEST

Reference: Schinck, G.H., Ozolins, M., Complexometric Determination of Aromatic Hydrocarbons with Tetracyanoethylene, Anal. Chem., 33, 11, 1961.

Range: 0-15000 mg/kg

Common Synonyms: Cumol, Isopropylbenzene, 2-Phenylpropane.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of respiratory tract, sore throat, coughing, shortness of breath, nausea. Ingestion- abdominal pain, vomiting. Contact - skin- redness and irritation.
Toxicology: Moderately toxic by all routes.
TLV - (skin) 50 ppm; 245 mg/cubic meter

Environment:
Fish Toxicity - 110 ppm/24h/brine shrimp/TLm/saltwater.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 250 mL Erlenmeyer Flasks
2 x 150 mL Beakers
2 x 50 mL Stoppered Graduated Mixing Cylinders
2 x 25 mL Graduated Cylinders
1 x 200 mL Volumetric Flask
5 x 5 mL Disposable Pipets
1 x 100 mL Beaker
Timing Device
Whatman #1 Filter Paper
2 x Long-stemmed Funnels
Balance

REQUIRED REAGENTS

0.1 M Tetracyanoethylene Solution
Chloroform

Preparation of 0.1 M Tetracyanoethylene solution -

Dissolve 2.562 g of tetracyanoethylene in 10 mL of acetone in a 100 mL beaker. Transfer the solution to a 200 mL volumetric flask with methylene chloride. Dilute to the 200 mL mark with methylene chloride. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of chloroform to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Transfer 20 mL of each filtrate to separate 50 mL mixing cylinders.

6. Pipet 5 mL of the tetracyanoethylene solution into each mixing cylinder, stopper, and mix well. Wait 2-3 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the spectrophotometer to read 440 nm.

8. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

9. Replace the Zero Scale card with the CUMENE in SOIL card.

10. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

11. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

12. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Cumene in Soil concentration from the card and record this result.

CUMENE IN WATER AND WASTEWATER

TETRACYANOETHYLENE COLORIMETRIC TEST

Reference: Schinck, G.H., Ozolins, M., Complexometric Determination of Aromatic Hydrocarbons with Tetracyanoethylene, Anal. Chem., 33, 11, 1961.

Range: 0-15000 mg/L

Common Synonyms: Cumol, Isopropylbenzene, 2-Phenylpropane.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of respiratory tract, sore throat, coughing, shortness of breath, nausea. Ingestion- abdominal pain, vomiting. Contact - skin- redness and irritation.
Toxicology: Moderately toxic by all routes.
TLV - (skin) 50 ppm; 245 mg/cubic meter

Environment:
Fish Toxicity - 110 ppm/24h/brine shrimp/TLm/saltwater.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 150 mL Beakers
2 x 125 mL Separatory Funnels
2 x 50 mL Stoppered Graduated Mixing Cylinders
2 x 25 mL Graduated Cylinders
1 x 200 mL Volumetric Flask
5 x 5 mL Disposable Pipets
1 x 100 mL Beaker
Timing Device
Balance

REQUIRED REAGENTS

0.1 M Tetracyanoethylene Solution
Chloroform

Preparation of 0.1 M Tetracyanoethylene solution -

Dissolve 2.562 g of tetracyanoethylene in 10 mL of acetone in a 100 mL beaker. Transfer the solution to a 200 mL volumetric

flask with methylene chloride. Dilute to the 200 mL mark with methylene chloride. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 25 mL water sample in a graduated cylinder. Pour the sample into a clean 125 mL separatory funnel. Label the funnel BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 25 mL mark. Pour the water into a second clean 125 mL separatory funnel. Label the funnel TEST.

3. Pipet 25 mL of chloroform into each of the 125 mL separatory funnels, stopper them, and shake well. Invert the separatory funnel periodically and open the stopcock to vent any buildup of gases.

4. Allow the phases to separate (usually 2-3 minutes).

5. Drain from each funnel 25 mL of the bottom (chloroform) layer into separate graduated mixing cylinders.

6. Pipet 5 mL of the tetracyanoethylene solution into each mixing cylinder, stopper, and mix well. Wait 2-3 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

5. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

6. Replace the Zero Scale card with the CUMENE in WATER card.

7. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

8. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

9. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Cumene in Water concentration from the card and record this result.

ETHANOL IN SOIL

CERIC AMMONIUM NITRATE METHOD

Reference: Reid, V.W., Truelove, R.K., The Colorimetric Determination of Alcohols, The Analyst, Vol. 77, 325, 1952.

Range: 2000 - 35000 mg/kg

Common Synonyms: Anhydrous Alcohol, Ethyl Alcohol, Alcohol, grain alcohol, Methyl Carbinol, Ethyl Hydrate.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation and Ingestion - nausea, vomiting, flushing, mental excitement or depression, impaired perception, uncoordination, stupor, coma. Contact - skin - drying and dermatitis; eyes - irritation and watering.
Toxicology: Low toxicity by all routes.

Environment:
Aquatic Toxicity Rating - >1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer flasks
2 x funnels
filter paper
2 x 150 mL beakers
50 mL graduated cylinder
Hach spectrophotometer
2 x cuvettes
graduated 5 mL pipet

REQUIRED REAGENTS

Ceric Ammonium Nitrate Solution

Preparation of Ceric ammonium nitrate solution -

Dissolve 43.27 grams of Ceric ammonium nitrate powder in 100 mL of deionized water in a 150 mL beaker. When fully dissolved, transfer to a 100 mL reagent bottle with screwtop lid.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Add 2 mL of Ceric ammonium nitrate solution to each beaker.

6. Adjust the Wavelength Dial of the spectrophotometer to read 460 nm.

7. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

8. Replace the Zero Scale card with the ETHANOL in SOIL card.

9. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

10. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

11. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Ethanol concentration from the card and record this result.

ETHANOL IN WATER AND WASTEWATER

CERIC AMMONIUM NITRATE METHOD

Reference: Reid, V.W., Truelove, R.K., The Colorimetric Determination of Alcohols, The Analyst, Vol. 77, 325, 1952.

Range: 200 - 10000 mg/L

Common Synonyms: Anhydrous Alcohol, Ethyl Alcohol, Alcohol, grain alcohol, Methyl Carbinol, Ethyl Hydrate.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation and Ingestion - nausea, vomiting, flushing, mental excitement or depression, impaired perception, uncoordination, stupor, coma. Contact - skin - drying and dermatitis; eyes - irritation and watering.
Toxicology: Low toxicity by all routes.

Environment:
Aquatic Toxicity Rating - >1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 150 mL beakers
50 mL graduated cylinder
Hach spectrophotometer
2 x cuvettes
graduated 5 mL pipet

REQUIRED REAGENTS

Ceric Ammonium Nitrate Solution

Preparation of Ceric ammonium nitrate solution -

Dissolve 43.27 grams of Ceric ammonium nitrate powder in 100 mL of deionized water in a 150 mL beaker. When fully dissolved, transfer to a 100 mL reagent bottle with screwtop lid.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 150 mL beaker. Label the beaker BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 150 mL beaker. Label the beaker TEST.

3. Add 2 mL of Ceric ammonium nitrate solution to each beaker.

4. Adjust the Wavelength Dial of the spectrophotometer to read 450 nm.

5. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

6. Replace the Zero Scale card with the ETHANOL in WATER card.

7. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

8. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

9. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Ethanol concentration from the card and record this result.

ETHANOLAMINE IN WATER AND WASTEWATER

PERIODIC ACID TEST METHOD

Reference: Lehman, A.J., Newman, H.W., J. Pharmacol. Exptl. Therap., 60, 312, 1937.

Range: 0 - 500 mg/L

Common Synonyms: MEA. Monoethanolamine, 2-Aminoethanol, Ethyolamine, Colamine, Glycinol.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation and coughing. Ingestion- irritation, nasea, vomiting. Contact: skin- irritation; eyes- irritation, possible corneal injury.
Toxicology: Moderately toxic by all routes
TLV - (inhalation) 3 ppm; 8 mg/cubic meter

Environment:
Aquatic Toxicity Rating: 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 250 mL Erlenmeyer flasks
- 25 mL buret
- Battery operated magnetic stirrer
- 2 x magnetic stirring bars
- 50 mL graduated cylinder
- 1 x 100 mL volumetric flask
- 1 x 500 mL volumetric flask
- 2 x 1000 mL volumetric flask
- 2 x 50 mL beakers

REQUIRED REAGENTS

- 0.5 M Periodic Acid
- 0.1 N Arsenious Acid
- 0.1 N Iodine
- 15% Potassium Iodide solution
- Saturated Sodium Bicarbonate solution
- Starch Indicator solution
- Arsenic trioxide (for standardization of Iodine)

Preparation of Periodic Acid solution, 0.5 M

Dissolve 11.397 grams of periodic acid in 50 mL of deionized water in a 150 mL beaker. Add 6.5 mL of concentrated sulfuric acid to the beaker. Transfer the solution to a 500 mL volumetric flask and dilute to the 500 mL mark with deionized water.

Preparation of Saturated Sodium Bicarbonate solution -

Add 20 g of sodium bicarbonate to a 250 mL beaker containing 100 mL of deionized water. NOTE - not all bicarbonate will dissolve.

Preparation of 15% Potassium Iodide -

Dissolve 15.000 g of potassium iodide in 50 mL of deionized water in a 100 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

Preparation of Starch solution -

Dissolve 1.0 g of soluble starch in 500 mL of hot (preferably boiling) deionized water.

Preparation of 0.1 N Iodine solution -

Dissolve 12.69 g of iodine in a 250 mL beaker containing 40 g of potassium iodide and 25 mL of deionized water. Stir to dissolve the iodine. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water. Mix the solution thoroughly.

Preparation of 0.1 N Arsenious Acid solution -

Dissolve 5.000 g of Sodium Arsenite in a 50 mL beaker containing 3.0 g of sodium hydroxide and 10 mL of deionized water. Allow the solution to stand, swirling it occasionally until the solid has completely dissolved. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water. Mix the solution thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Put a magnetic stir bar in each of the two flasks.

4. Add 5.0 mL of 0.5 M Periodic Acid to each flask. Put one of the flasks on the magnetic stirrer. After 10 minutes, switch the flasks and stir the other flask for 10 minutes for a total reaction time of 20 minutes.

5. Add 10 mL of saturated Sodium Bicarbonate to each flask. Then add 0.5 mL of Potassium Iodide solution to each flask.

6. Add 20 mL of Arsenious Acid solution to each sample.

7. Add approximately 3 grams of Sodium Bicarbonate to each flask and stir well.

8. Fill the burette with 0.1N Iodine and record burette reading. Add 5 mL of Starch indicator solution to the BLANK sample and titrate with 0.1N Iodine until the blue colour remains stable (1 minute). Record final burette reading.

9. Refill burette and record burette reading. Add 5 mL of Starch indicator solution to the TEST sample and titrate until the blue colour remains stable (1 minute). Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Ethanolamine by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x (titrant concentration) x 63.4]

where titrant concentration is 0.1 unless standard Iodine solution is not used.

ETHYLBENZENE IN SOILS

TETRACYANOETHYLENE COLORIMETRIC TEST

Reference: Schinck, G.H., Ozolins, M., Complexometric Determination of Aromatic Hydrocarbons with Tetracyanoethylene, Anal. Chem., 33, 11, 1961.

Range: 0-10000 mg/kg

Common Synonyms: Phenylethane, EB, Ethylbenzol.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of mucous membranes, headache, dizziness, narcosis, and coma. Contact - skin- irritation, defatting and dermatitis; eyes- irritation and burning. Ingestion - symptoms similar to inhalation.
Toxicology: Moderately toxic by all routes.
TLV - (inhalation) 100 ppm; 435 mg/cubic meter
Short-term Inhalation Limits - 125 ppm; 545 mg/cubic meter (15 min).

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 250 mL Erlenmeyer Flasks
2 x 150 mL Beakers
2 x 50 mL Stoppered Graduated Mixing Cylinders
2 x 25 mL Graduated Cylinders
1 x 200 mL Volumetric Flask
5 x 5 mL Disposable Pipets
1 x 100 mL Beaker
Timing Device
Whatman #1 Filter Paper
2 x Long-stemmed Funnels
Balance

REQUIRED REAGENTS

0.1 M Tetracyanoethylene Solution
Chloroform

Preparation of 0.1 M Tetracyanoethylene solution -

Dissolve 2.562 g of tetracyanoethylene in 10 mL of acetone in a

100 mL beaker. Transfer the solution to a 200 mL volumetric flask with methylene chloride. Dilute to the 200 mL mark with methylene chloride. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of chloroform to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Transfer 20 mL of each filtrate to separate 50 mL mixing cylinders.

6. Pipet 5 mL of the tetracyanoethylene solution into each mixing cylinder, stopper, and mix well. Wait 2-3 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the spectrophotometer to read 440 nm.

8. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

9. Replace the Zero Scale card with the ETHYLBENZENE in SOIL card.

10. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

11. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

9. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Ethylbenzene in Soil concentration from the card and record this result.

ETHYLBENZENE IN WATER AND WASTEWATER

TETRACYANOETHYLENE COLORIMETRIC TEST

Reference: Schinck, G.H., Ozolins, M., Complexometric Determination of Aromatic Hydrocarbons with Tetracyanoethylene, Anal. Chem., 33, 11, 1961.

Range: 0-750 mg/L

Common Synonyms: Phenylethane, EB, Ethylbenzol.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of mucous membranes, headache, dizziness, narcosis, and coma. Contact - skin- irritation, defatting and dermatitis; eyes- irritation and burning. Ingestion - symptoms similar to inhalation.
Toxicology: Moderately toxic by all routes.
TLV - (inhalation) 100 ppm; 435 mg/cubic meter
Short-term Inhalation Limits - 125 ppm; 545 mg/cubic meter (15 min).

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 150 mL Beakers
2 x 125 mL Separatory Funnels
2 x 50 mL Stoppered Graduated Mixing Cylinders
2 x 25 mL Graduated Cylinders
1 x 200 mL Volumetric Flask
5 x 5 mL Disposable Pipets
1 x 100 mL Beaker
Timing Device
Balance

REQUIRED REAGENTS

0.1 M Tetracyanoethylene Solution
Chloroform

Preparation of 0.1 M Tetracyanoethylene solution -

Dissolve 2.562 g of tetracyanoethylene in 10 mL of acetone in a 100 mL beaker. Transfer the solution to a 200 mL volumetric flask with methylene chloride. Dilute to the 200 mL mark with

methylene chloride. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 25 mL water sample in a graduated cylinder. Pour the sample into a clean 125 mL separatory funnel. Label the funnel BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 25 mL mark. Pour the water into a second clean 125 mL separatory funnel. Label the funnel TEST.

3. Pipet 25 mL of chloroform into each of the 125 mL separatory funnels, stopper them, and shake well. Invert the separatory funnel periodically and open the stopcock to vent any buildup of gases.

4. Allow the phases to separate (usually 2-3 minutes).

5. Drain from each funnel 25 mL of the bottom (chloroform) layer into separate graduated mixing cylinders.

6. Pipet 5 mL of the tetracyanoethylene solution into each mixing cylinder, stopper, and mix well. Wait 2-3 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

8. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

9. Replace the Zero Scale card with the ETHYLBENZENE in WATER card.

10. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

11. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

12. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Ethylbenzene in Water concentration from the card and record this result.

ETHYLENE GLYCOL IN SOIL

PERIODIC ACID TEST METHOD

Reference: Lehman, A.J., Newman, H.W., J. Pharmacol. Exptl. Therap., 60, 312, 1937.

Range: 100 - 3000 mg/kg

Common Synonyms: Antifreeze, Glycol, Monoethylene Glycol, Ethylene Dihydrate, 1,2-Ethanediol.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation: intoxication, headache. Ingestion: intoxication, headache, vomiting, cyanosis, unconsciousness with convulsions. Contact: Skin- absorbed causing intoxication; Eyes- irritation.
Toxicology: Moderately toxic by all routes

Environment:
Aquatic Toxicity Rating: 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer Flasks
2 x Funnels
2 x 150 mL Beakers
2 x Magnetic Stirring Bars
50 mL Graduated Cylinder
1 x 25 mL Burette
Whatman #1 Filter Paper
Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

0.1M Periodic Acid
0.1N Arsenious Acid
0.1N Iodine
15% Potassium Iodide solution
Saturated Sodium Bicarbonate solution
Starch Indicator solution
Arsenic trioxide (for standardization of Iodine)

Preparation of Periodic Acid solution, 0.1M

Dissolve 2.280 grams of periodic acid in 50 mL of deionized water in a 150 mL beaker. Add 3 mL of concentrated sulfuric acid to the beaker. Transfer the solution to a 500 mL volumetric flask and dilute to the 500 mL mark with deionized water.

Preparation of Saturated Sodium Bicarbonate solution -
Add 20 g of sodium bicarbonate to a 250 mL beaker containing 100 mL of deionized water. NOTE - not all bicarbonate will dissolve.

Preparation of 15% Potassium Iodide -

Dissolve 15.000 g of potassium iodide in 50 mL of deionized water in a 100 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

Preparation of Starch solution -

Dissolve 1.0 g of soluble starch in 500 mL of hot (preferably boiling) deionized water.

Preparation of 0.1 N Iodine solution -

Dissolve 12.69 g of iodine in a 250 mL beaker containing 40 g of potassium iodide and 25 mL of deionized water. Stir to dissolve the iodine. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water. Mix the solution thoroughly.

Preparation of 0.1 N Arsenious Acid solution -

Dissolve 5.000 g of Sodium Arsenite in a 50 mL beaker containing 3.0 g of sodium hydroxide and 10 mL of deionized water. Allow the solution to stand, swirling it occasionally until the solid has completely dissolved. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water. Mix the solution thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Put a magnetic stir bar in each of the two flasks.

4. Add 5.0 mL of 0.1M Periodic Acid to each flask. Put one of the flasks on the magnetic stirrer. After 10 minutes, switch the flasks and stir the other flask for 10 minutes for a total reaction time of 20 minutes.

5. Add 10 mL of saturated Sodium Bicarbonate to each flask. Then add 0.5 mL of Potassium Iodide solution to each flask.

6. Add 20 mL of Arsenious Acid solution to each sample.

7. Add approximately 3 grams of Sodium Bicarbonate to each flask and stir well.

8. Fill the burette with 0.1N Iodine and record burette reading. Add 5 mL of Starch indicator solution to the BLANK sample and titrate with 0.1N Iodine until the blue colour remains stable (1 minute). Record final burette reading.

9. Refill burette and record burette reading. Add 5 mL of Starch indicator solution to the TEST sample and titrate until the blue colour remains stable (1 minute). Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Ethylene Glycol by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x (titrant concentration) x 3400]

where titrant concentration is 0.1 unless standard Iodine solution is not used.

ETHYLENE GLYCOL IN WATER AND WASTEWATER

PERIODIC ACID TEST METHOD

Reference: Lehman, A.J., Newman, H.W., J. Pharmacol. Expt1. Therap., 60, 312, 1937.

Range: 0 - 500 mg/L

Common Synonyms: Antifreeze, Glycol, Monoethylene Glycol, Ethylene Dihydrate, 1,2-Ethanediol.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation: intoxication, headache. Ingestion: intoxication, headache, vomiting, cyanosis, unconsciousness with convulsions. Contact: Skin- absorbed causing intoxication; Eyes- irritation.
Toxicology: Moderately toxic by all routes

Environment:
Aquatic Toxicity Rating: 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 250 mL Erlenmeyer flasks
- 25 mL buret
- Battery operated magnetic stirrer
- 2 x magnetic stirring bars
- 50 mL graduated cylinder
- 1 x 100 mL volumetric flask
- 1 x 500 mL volumetric flask
- 2 x 1000 mL volumetric flask
- 2 x 50 mL beakers

REQUIRED REAGENTS

- 0.5 M Periodic Acid
- 0.1 N Arsenious Acid
- 0.1 N Iodine
- 15% Potassium Iodide solution
- Saturated Sodium Bicarbonate solution
- Starch Indicator solution
- Arsenic trioxide (for standardization of Iodine)

Preparation of Periodic Acid solution, 0.5 M

Dissolve 11.397 grams of periodic acid in 50 mL of deionized water in a 150 mL beaker. Add 6.5 mL of concentrated sulfuric acid to the beaker. Transfer the solution to a 500 mL volumetric flask and dilute to the 500 mL mark with deionized water.

Preparation of Saturated Sodium Bicarbonate solution -

Add 20 g of sodium bicarbonate to a 250 mL beaker containing 100 mL of deionized water. NOTE - not all bicarbonate will dissolve.

Preparation of 15% Potassium Iodide -

Dissolve 15.000 g of potassium iodide in 50 mL of deionized water in a 100 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

Preparation of Starch solution -

Dissolve 1.0 g of soluble starch in 500 mL of hot (preferably boiling) deionized water.

Preparation of 0.1 N Iodine solution -

Dissolve 12.69 g of iodine in a 250 mL beaker containing 40 g of potassium iodide and 25 mL of deionized water. Stir to dissolve the iodine. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water. Mix the solution thoroughly.

Preparation of 0.1 N Arsenious Acid solution -

Dissolve 5.000 g of Sodium Arsenite in a 50 mL beaker containing 3.0 g of sodium hydroxide and 10 mL of deionized water. Allow the solution to stand, swirling it occasionally until the solid has completely dissolved. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water. Mix the solution thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Put a magnetic stir bar in each of the two flasks.

4. Add 5.0 mL of 0.1M Periodic Acid to each flask. Put one of the flasks on the magnetic stirrer. After 10 minutes, switch the flasks and stir the other flask for 10 minutes for a total reaction time of 20 minutes.

5. Add 10 mL of saturated Sodium Bicarbonate to each flask. Then add 0.5 mL of Potassium Iodide solution to each flask.

6. Add 20 mL of Arsenious Acid solution to each sample.

7. Add approximately 3 grams of Sodium Bicarbonate to each flask and stir well.

8. Fill the burette with 0.1N Iodine and record burette reading. Add 5 mL of Starch indicator solution to the BLANK sample and titrate with 0.1N Iodine until the blue colour remains stable (1 minute). Record final burette reading.

9. Refill burette and record burette reading. Add 5 mL of Starch indicator solution to the TEST sample and titrate until the blue colour remains stable (1 minute). Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Ethylene Glycol by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x (titrant concentration) x 67.5]

where titrant concentration is 0.1 unless standard Iodine solution is not used.

FERRIC CHLORIDE IN SOILS

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/kg

Common Synonyms: Iron Chloride, Ferric Trichloride, Ferric Perchloride, Iron Trichloride, Iron Perchloride, Iron (III) Chloride.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - nose and throat irritation. Ingestion- may cause burns of the mucous membranes and severe irritation of the gastrointestinal tract. Contact - eyes- irritation; skin- irritation, dermatitis, staining, and burning.
Toxicology: Moderately to highly toxic by ingestion. Highly toxic by contact.
TLV - 1 mg/cubic meter (as Fe)
Short-term Inhalation Limits - 2 mg/cubic meter (as Fe) (15 min)

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 2 x long-stem funnels
- Whatman 12.5 cm #1 Filter Paper
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 1 x 100 mL Volumetric Flask
- 2 x 50 mL Beakers
- 4 x 150 mL Beakers
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer
- pH Meter and Electrode

REQUIRED REAGENTS

- 0.1 N Nitric Acid solution
- 0.014 N Mercuric Nitrate solution
- Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Pipet 25 mL of each filtrate into two more separate, clean 150 mL beakers. Add 75 mL of deionized water to each using the graduated cylinder.

6. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

7. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

8. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

9. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

10. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Ferric Chloride by solving the following equation:

$$\text{Concentration (mg/kg)} = \frac{[\ (\text{adjusted titrant volume}) \times 305\ ]}{}$$

Other Ferric Chloride compounds -

$FeCl_3$ - Concentration (mg/kg) = [a.t.v. x 305]

$FeCl_3 \cdot 2.5\ H_2O$ - Concentration (mg/kg) = [a.t.v. x 390]

$FeCl_3 \cdot 6\ H_2O$ - Concentration (mg/kg) = [a.t.v. x 508]

FERRIC CHLORIDE IN WATER AND WASTEWATER

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/L

Common Synonyms: Iron Chloride, Ferric Trichloride, Ferric Perchloride, Iron Trichloride, Iron Perchloride, Iron (III) Chloride.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - nose and throat irritation. Ingestion- may cause burns of the mucous membranes and severe irritation of the gastrointestinal tract. Contact - eyes- irritation; skin- irritation, dermatitis, staining, and burning.
Toxicology: Moderately to highly toxic by ingestion. Highly toxic by contact.
TLV - 1 mg/cubic meter (as Fe)
Short-term Inhalation Limits - 2 mg/cubic meter (as Fe) (15 min)

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 1 x 100 mL Volumetric Flask
- 2 x 50 mL Beakers
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

- 0.1 N Nitric Acid solution
- 0.014 N Mercuric Nitrate solution
- Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

4. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

5. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

6. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

7. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

8. Refill the burette. Record initial burette reading. Titrate

the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Ferric Chloride by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 15.25]

Other Ferric Chloride compounds -

$FeCl_3$ - Concentration (mg/L) = [a.t.v. x 15.25]

$FeCl_3 \cdot 2.5\ H_2O$ - Concentration (mg/L) = [a.t.v. x 19.48]

$FeCl_3 \cdot 6\ H_2O$ - Concentration (mg/L) = [a.t.v. x 25.41]

FORMIC ACID IN WATER

SODIUM HYDROXIDE TITRATION METHOD

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0 - 10000 mg/L

Common Synonyms: Methenoic Acid, Formylic Acid, Hydrogen Carboxylic Acid.

HAZARDOUS DATA

Human Health
Symptoms: Contact - irritation and burns to skin and eyes. Inhalation - irritation of respiratory tract, coughing, bronchitis, difficulty breathing. Ingestion - nausea, vomiting, dizziness, unconsciousness.
Toxicology: Moderately toxic upon inhalation, ingestion and contact.
TLV - 5 ppm; 9 mg/cubic meter

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 50 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
1 x 25 mL Burette

REQUIRED REAGENTS

Phenolphthalein indicator solution
0.02N Sodium Hydroxide solution
*Note: All required reagents supplied by Hach company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the

flask TEST.

3. Fill the 0 - 25 mL burette with 0.02N Sodium Hydroxide solution and record the initial burette reading.

4. Add 6 drops of phenolphthalein solution to each flask.

5. Titrate the BLANK water sample against the sodium hydroxide solution; add slowly, with continuous swirling until a pink colour appears for more than 30 seconds.

6. Record the final burette reading.

7. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Formic Acid by solving the following equation:

$$\text{Concentration (mg/L)} = [\ (\text{adjusted titrant volume}) \times 20.3\ ]$$

HYDROCHLORIC ACID IN SOILS

MERCURIC NITRATE TITRATION METHOD

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0 - 25000 mg/kg

Common Synonyms: Muriatic Acid, Chlorohydric Acid.

HAZARDOUS DATA

Human Health
Symptoms: Contact - eyes- severe irritation, burning, visual impairment may result; skin- can cause serious burns. Inhalation - vapours can produce severe irritation of the upper respiratory tract. Ingestion - very corrosive, can cause serious internal injury.
Toxicology: Moderately toxic upon inhalation, ingestion and contact.
TLV - 5 ppm; 7 mg/cubic meter

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 2 x Long-Stem Funnels
- Whatman 12.5 cm #1 Filter Paper
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 1 x 100 mL Volumetric Flask
- 2 x 50 mL Beakers
- 4 x 150 mL Beakers
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer
- pH Meter and Electrode

REQUIRED REAGENTS

- 0.1 N Nitric Acid solution
- 0.014 N Mercuric Nitrate solution
- Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Pipet 25 mL of each filtrate into two more separate, clean 150 mL beakers. Add 75 mL of deionized water to each using the graduated cylinder.

6. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

7. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

8. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

9. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

10. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Hydrochloric Acid by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x 206]

HYDROCHLORIC ACID IN WATER AND WASTEWATER

MERCURIC NITRATE TITRATION METHOD

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0 - 25000 mg/L

Common Synonyms: Muriatic Acid, Chlorohydric Acid.

HAZARDOUS DATA

Human Health
Symptoms: Contact - eyes- severe irritation, burning, visual impairment may result; skin- can cause serious burns. Inhalation - vapours can produce severe irritation of the upper respiratory tract. Ingestion - very corrosive, can cause serious internal injury.
Toxicology: Moderately toxic upon inhalation, ingestion and contact.
TLV - 5 ppm; 7 mg/cubic meter

EXPERIMENTAL REQUIREMENTS

Apparatus:
1 x 100 mL Graduated Cylinder
2 x 250 mL Erlenmeyer Flasks
1 x 25 mL Burette
1 x 1000 mL Volumetric Flask
1 x 100 mL Volumetric Flask
2 x 50 mL Beakers
Magnetic Stir Bars
Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

0.1 N Nitric Acid solution
0.014 N Mercuric Nitrate solution
Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

4. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

5. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

6. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

7. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

8. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Hydrochloric Acid by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 38.92]

HYDROCHLORIC ACID IN WATER AND WASTEWATER

SODIUM HYDROXIDE TITRATION METHOD

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0 - 10000 mg/L

Common Synonyms: Muriatic Acid, Chlorohydric Acid.

HAZARDOUS DATA

Human Health
Symptoms: Contact - eyes- severe irritation, burning, visual impairment may result; skin- can cause serious burns. Inhalation - vapours can produce severe irritation of the upper respiratory tract. Ingestion - very corrosive, can cause serious internal injury.
Toxicology: Moderately toxic upon inhalation, ingestion and contact.
TLV - 5 ppm; 7 mg/cubic meter

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 50 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
1 x 25 mL Burette

REQUIRED REAGENTS

Phenolphthalein indicator solution
0.02N Sodium Hydroxide solution
*Note: All required reagents supplied by Hach company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the

flask TEST.

3. Fill the 0 - 25 mL burette with 0.02N Sodium Hydroxide solution and record the initial burette reading.

4. Add 6 drops of phenolphthalein solution to each flask.

5. Titrate the BLANK water sample against the sodium hydroxide solution; add slowly, with continuous swirling until a pink colour appears for more than 30 seconds.

6. Record the final burette reading.

7. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Hydrochloric Acid by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 38.92]

HYDROGEN PEROXIDE IN WATER AND WASTEWATER

o-TOLIDINE COLORIMETRIC TEST

Reference: Feigel, F., Spot Tests in Organic Analysis, Elesevier Publishing, 1966.

Range: 0-10 mg/L

Common Synonyms: Peroxide, Hydrogen Dioxide, Hydroperoxide.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - sore throat, coughing, laboured breathing, headache, nausea, weakness. Ingestion - pain in swallowing, intense thirst, nausea and vomiting, difficulty breathing, convulsions, coma. Contact - skin- burning, painful blistering, shock; eyes- stinging, burning.
Toxicology: Highly toxic by all routes.
TLV - (inhalation) 1 ppm; 1.5 mg/cubic meter
Short-term Exposure Limits - 2 ppm; 3 mg/cubic meter

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
2 x 100 ml Volumetric Flasks
2 x 50 mL beaker
Graduated 1 mL Disposable Pipets

REQUIRED REAGENTS

1% o-Tolidine solution
1% Ferrous Sulfate solution
Sodium Acetate Buffer solution

Preparation of 1% o-Tolidine solution -

Dissolve 1.000 g of o-Tolidine in 25 mL of ethanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with ethanol. Mix thoroughly.

Preparation of 1% Ferrous Sulfate solution -

Dissolve 1.000 g of Ferrous Sulfate in 50 mL of deionized water in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

Preparation of Sodium Acetate Buffer solution -

Use pH Buffer 4 and/or 4.01 Sodium Acetate Buffer solution (commercially available).

PROCEDURE

1. From an uncontaminated source in the area obtain a 100 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 100 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. To each of the Erlenmeyer flasks, add 1 mL of the 1% o-Tolidine solution with a graduated pipet. Mix well.

4. Then, add 1 mL of the 1% ferrous sulfate solution to both flasks. Mix thoroughly.

5. To each flask, add by pipet 1 mL of the Buffer solution and mix.

6. Adjust the Wavelength Dial of the spectrophotometer to read 450 nm.

7. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

8. Replace the Zero Scale card with the HYDROGEN PEROXIDE in WATER card.

7. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

8. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

9. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Hydrogen Peroxide concentration from the card and record this result.

HYDROGEN PEROXIDE IN WATER AND WASTEWATER

SODIUM THIOSULFATE TITRIMETRIC TEST

Reference: Treadwell, J., Hall, K., Analytical Chemistry, Vol II, Quantitative Analysis, 8th Ed., 1935.

Range: 0-75 mg/L

Common Synonyms: Peroxide, Hydrogen Dioxide, Hydroperoxide.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - sore throat, coughing, laboured breathing, headache, nausea, weakness. Ingestion - pain in swallowing, intense thirst, nausea and vomiting, difficulty breathing, convulsions, coma. Contact - skin- burning, painful blistering, shock; eyes- stinging, burning.
Toxicology: Highly toxic by all routes.
TLV - (inhalation) 1 ppm; 1.5 mg/cubic meter
Short-term Exposure Limits - 2 ppm; 3 mg/cubic meter

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 100 mL Graduated Cylinders
- 2 x 250 mL Erlenmeyer Flasks
- 1 x 1000 ml Volumetric Flasks
- 2 x 400 mL Beakers
- 2 x 250 mL Beakers
- 1 x 25 mL Burette
- 2 x Magnetic Stir Bars
- Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

- 10% Potassium Iodide solution
- 0.02 N Sodium Thiosulfate solution

for standardization -
- Potassium Dichromate
- Sulfuric Acid, concentrated
- Sodium Carbonate
- Potassium Iodide
- Starch indicator solution

Preparation of 10% Potassium Iodide solution -

To a 500 mL beaker containing 400 mL of deionized water, slowly add 25 mL of concentrated sulfuric acid. Allow the solution to cool and dilute to 500 mL with deionized water. Add 50.0 g of potassium iodide to the acidic solution and dissolve. Mix thoroughly.

Preparation of 0.02 N Sodium Thiosulfate solution -

Dissolve 5.000 g of sodium thiosulfate pentahydrate in 1 L of recently boiled and cooled deionized water. Add 0.2 g of sodium bicarbonate as a preservative.

Standardization of 0.02 N Sodium Thiosulfate -

Weigh accurately three portions of pure, dry potassium dichromate, each weighing approximately 0.04 g, into 500 mL Erlenmeyer flasks. Dissolve each portion with 100 mL of deionized water and 4 mL of concentrated sulfuric acid.

To the first sample, carefully add 2 g of sodium carbonate with gentle swirling to liberate carbon dioxide. Then add 5 g of potassium iodide dissolved in 5 mL of deionized water, swirl, cover with a watch glass, and allow the solution to stand for 3 minutes. Dilute the solution to 200 mL with deionized water.

Titrate the potassium dichromate solution with the sodium thiosulfate solution umtil the yellowish (iodine) colour has nearly disappeared. Add 5 mL of the starch indicator solution (from Ethylene Glycol reagent methods) and continue titrating until the blue colour disappears.

Repeat the standardization process for the other two samples of potassium dichromate. Calculate the concentration of the sodium thiosulfate solution by mole/volume ratio and average the three results for a mean concentration.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Measure 50 mL of 10% potassium iodide solution into a graduated cylinder. Pour the potassium iodide solution into the BLANK flask.

4. Fill the 0-25 mL burette with standardizid Sodium Thiosulfate solution and record the initial burette reading.

5. Add a magnetic stir bar to the flask, place the BLANK solution on the magnetic stirrer and turn ON.

6. Titrate the BLANK solution by slowly adding the sodium thiosulfate until the yellow colour persistently disappears. Record the final volume.

7. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Hydrogen Peroxide by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x (titrant concentration) x 160.6]

where titrant concentration is 0.02 unless standardization has been done.

ISOPROPANOL IN SOIL

CERIC AMMONIUM NITRATE METHOD

Reference: Reid, V.W., Truelove, R.K., The Colorimetric Determination of Alcohols, The Analyst, Vol. 77, 325, 1952.

Range: 2000 - 50000 mg/kg

Common Synonyms: Isopropyl Alcohol, 2-Propanol, Secondary Propyl Alcohol, Dimethyl Carbinol, Petrohol, Rubbing Alcohol.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation and Ingestion - headache, dizziness, flushing, mental depression, nausea, vomiting, narcosis.
Contact - eyes - severely irritating; skin - defatting and cracking.
Toxicology: Low toxicity by contact. Moderately toxic by ingestion.

Environment:
Aquatic Toxicity Rating - 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer flasks
2 x funnels
filter paper
2 x 150 mL beakers
50 mL graduated cylinder
Hach spectrophotometer
2 x cuvettes
graduated 5 mL pipet

REQUIRED REAGENTS

Ceric Ammonium Nitrate Solution

Preparation of Ceric ammonium nitrate solution -

Dissolve 43.27 grams of Ceric ammonium nitrate powder in 100 mL of deionized water in a 150 mL beaker. When fully dissolved, transfer to a 100 mL reagent bottle with screwtop lid.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Add 2 mL of Ceric ammonium nitrate solution to each beaker.

6. Adjust the Wavelength Dial of the spectrophotometer to read 460 nm.

7. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

8. Replace the Zero Scale card with the ISOPROPANOL in SOIL card.

9. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

10. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

11. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Isopropanol concentration from the card and record this result.

ISOPROPANOL IN WATER

CERIC AMMONIUM NITRATE METHOD

Reference: Reid, V.W., Truelove, R.K., The Colorimetric Determination of Alcohols, The Analyst, Vol. 77, 325, 1952.

Range: 200 - 10000 mg/L

Common Synonyms: Isopropyl Alcohol, 2-Propanol, Secondary Propyl Alcohol, Dimethyl Carbinol, Petrohol, Rubbing Alcohol.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation and Ingestion - headache, dizziness, flushing, mental depression, nausea, vomiting, narcosis. Contact - eyes - severely irritating; skin - defatting and cracking.
Toxicology: Low toxicity by contact. Moderately toxic by ingestion.

Environment:
Aquatic Toxicity Rating - 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 150 mL beakers
50 mL graduated cylinder
Hach spectrophotometer
2 x cuvettes
graduated 5 mL pipet

REQUIRED REAGENTS

Ceric Ammonium Nitrate Solution

Preparation of Ceric ammonium nitrate solution -

Dissolve 43.27 grams of Ceric ammonium nitrate powder in 100 mL of deionized water in a 150 mL beaker. When fully dissolved, transfer to a 100 mL reagent bottle with screwtop lid.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 150 mL beaker. Label the beaker BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 150 mL beaker. Label the beaker TEST.

3. Add 2 mL of Ceric ammonium nitrate solution to each beaker.

4. Adjust the Wavelength Dial of the spectrophotometer to read 450 nm.

5. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

6. Replace the Zero Scale card with the ISOPROPANOL in WATER card.

7. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

8. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

9. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Isopropanol concentration from the card and record this result.

MALEIC ANHYDRIDE IN SOIL

HYDROLYSIS AND SODIUM HYDROXIDE TITRATION

Reference: Smith, D.M., Bryant, W.M.D., J. Am. Chem. Soc., 58, 2452, 1936.

Range: 250 - 5000 mg/kg

Common Synonyms: 2,5-Furandione, cis-Butenedioic Anhydride, Toxilic Anhydride.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of throat and respiratory tract, bronchitis. Ingestion - abdominal pain, vomiting, nausea. Contact - readily absorbed through skin and eyes, serious burns.
Toxicology: Highly toxic on contact.

Environment:
Aquatic Toxicity Rating - 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 125 Erlenmeyer flasks
- 2 x long-stemmed funnels
- Whatman #1 filter paper
- 2 x 150 mL beaker
- 50 mL graduated cylinder
- 2 x magnetic stirring bars
- battery operated magnetic stirrer
- 25 mL burette

REQUIRED REAGENTS

- 1:1 Pyridine/Water solution
- 0.02N Sodium Hydroxide
- Thymol blue indicator solution

Preparation of 1:1 pyridine/water solution -

Combine 75 mL of deionized water with 75 mL of pyridine in a 500 mL bottle with a screw-top lid.

Preparation of Thymol blue indicator solution -

Dissolve 0.4 grams of Thymol blue in 100 mL of deionized water and store in a 150 mL plastic bottle with lid.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of pyridine/water solution to each flask. Mix well for 5 minutes.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Put a magnetic stirring bar in each beaker. Add 6 drops of Thymol blue indicator to each sample.

6. Fill the burette with 0.02N Sodium Hydroxide solution. Record the initial burette reading. Place the BLANK sample on the magnetic stirrer and turn stirrer on to low speed. Titrate the BLANK sample with Sodium Hydroxide until the solution changes colour from yellow to blue-green. Record the final burette reading.

7. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Maleic Anhydride by solving the following equation:

$$\text{Concentration (mg/kg)} = [\ (\text{adjusted titrant volume}) \times 128.5\ ]$$

MALEIC ANHYDRIDE IN WATER

SODIUM HYDROXIDE TITRATION

Reference: Smith, D.M., Bryant, W.M.D., J. Am. Chem. Soc., 58, 2452, 1936.

Range: 25 - 1000 mg/L

Common Synonyms: 2,5-Furandione, cis-Butenedioic anhydride, toxilic anhydride.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of throat and respiratory tract, bronchitis. Ingestion - abdominal pain, vomiting, nausea. Contact - readily absorbed through skin and eyes, serious burns.
Toxicology: Highly toxic on contact.

Environment:
Aquatic Toxicity Rating - 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer flasks
50 mL graduated cylinder
2 x magnetic stirring bars
magnetic stirrer
25 mL burette

REQUIRED REAGENTS

0.02N Sodium Hydroxide
Phenolphthalein indicator solution

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Put a magnetic stirring bar in each flask. Add 6 drops of Phenolphthalein indicator to each sample.

4. Fill the burette with 0.02N Sodium Hydroxide solution. Record the initial burette reading. Place the BLANK sample on the magnetic stirrer and turn stirrer on to low speed. Titrate the BLANK sample with Sodium Hydroxide until pink colour remains for at least 1 minute. Record the final burette reading.

5. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Maleic Anhydride by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 18.87]

METHANOL IN SOIL

CERIC AMMONIUM NITRATE METHOD

Reference: Reid, V.W., Truelove, R.K., The Colorimetric Determination of Alcohols, The Analyst, Vol. 77, 325, 1952.

Range: 2000 - 40000 mg/kg

Common Synonyms: Methyl Alcohol, Carbinol, Wood Spirit, Wood Alcohol, Methyl Hydroxide.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation and Ingestion - headache, fatigue, nausea, visual impairment (blindness), itching of skin, convulsions, dermatitis, circulatory collapse, respiratory failure. Contact-readily absorbed through skin.
Toxicology: Low toxicity by contact, moderate by other routes.

Environment:
Aquatic Toxicity Rating - >1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer flasks
2 x funnels
filter paper
2 x 150 mL beakers
50 mL graduated cylinder
Hach spectrophotometer
2 x cuvettes
graduated 5 mL pipet

REQUIRED REAGENTS

Ceric Ammonium Nitrate Solution

Preparation of Ceric ammonium nitrate solution -

Dissolve 43.27 grams of Ceric ammonium nitrate powder in 100 mL of deionized water in a 150 mL beaker. When fully dissolved, transfer to a 100 mL reagent bottle with screwtop lid.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Add 2 mL of Ceric ammonium nitrate solution to each beaker.

6. Adjust the Wavelength Dial of the spectrophotometer to read 450 nm.

7. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

8. Replace the Zero Scale card with the METHANOL in SOIL card.

9. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

10. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

11. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Methanol concentration from the card and record this result.

METHANOL IN WATER

CERIC AMMONIUM NITRATE METHOD

Reference: Reid, V.W., Truelove, R.K., The Colorimetric Determination of Alcohols, The Analyst, Vol. 77, 325, 1952.

Range: 500 - 10000 mg/L

Common Synonyms: Methyl Alcohol, Carbinol, Wood Spirit, Wood Alcohol, Methyl Hydroxide.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation and Ingestion - headache, fatigue, nausea, visual impairment (blindness), itching of skin, convulsions, dermatitis, circulatory collapse, respiratory failure. Contact-readily absorbed through skin.
Toxicology: Low toxicity by contact, moderate by other routes.

Environment:
Aquatic Toxicity Rating - >1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 150 mL beakers
50 mL graduated cylinder
Hach spectrophotometer
2 x cuvettes
graduated 5 mL pipet

REQUIRED REAGENTS

Ceric Ammonium Nitrate Solution

Preparation of Ceric ammonium nitrate solution -

Dissolve 43.27 grams of Ceric ammonium nitrate powder in 100 mL of deionized water in a 150 mL beaker. When fully dissolved, transfer to a 100 mL reagent bottle with screwtop lid.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 150 mL beaker. Label the beaker BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 150 mL beaker. Label the beaker TEST.

3. Add 2 mL of Ceric ammonium nitrate solution to each beaker.

4. Adjust the Wavelength Dial of the spectrophotometer to read 460 nm.

5. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

6. Replace the Zero Scale card with the METHANOL in WATER card.

7. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

8. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

9. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Methanol concentration from the card and record this result.

METHYL ETHYL KETONE IN SOILS

2,4-DINITROPHENYLHYDRAZINE METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Permagon Press, 1963.

Range: 0 - 500 mg/kg

Common Synonyms: MEK, 2-Butanone, Ethyl Methyl Ketone

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - coughing, shortness of breath, headache, dizziness, irritation of respiratory tract. Ingestion- irritation of respiratory tract, nausea, vomiting. Contact- skin- defatting and dermatitis; eyes- irritation and burning.
Toxicology: Highly toxic by ingestion, moderately toxic by contact.
TLV - (inhalation) 200 ppm; 590 mg/cubic meter
Short-term Inhalation Limits - 300 ppm; 885 mg/cubic meter (15 min).

Environment:
Fish toxicity: 5640 ppm/48h/bluegill/TLm /freshwater
Aquatic Toxicity Rating - > 1000 ppm/96h/TLm/freshwater.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
2 x 100 mL Mixing Cylinders
5 x 10 mL Disposable Pipets
2 x 150 mL Beakers
1 x 100 mL Volumetric Flask
Timing Device
Whatman #1 Filter Paper
2 x Long Stemmed Funnels
Balance

REQUIRED REAGENTS

Pyridine Stabilizer
33% Potassium Hydroxide Solution
2,4-Dinitrophenylhydrazine Solution

Preparation of Pyridine Stabilizer

Add 100 mL of deionized water to 400 mL of pyridine. Mix thoroughly. Store in a brown glass bottle.

Preparation of 33% Potassium Hydroxide Solution

Dissolve 33.0 g of potassium hydroxide in a 150 mL beaker containing 100 mL of methanol.

Preparation of 2,4-Dinitrophenylhydrazine solution

Dissolve 0.100 g of 2,4-Dinitrophenylhydrazine in a 100 mL beaker containing 50 mL of methanol and 4 mL of concentrated hydrochloric acid. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Transfer 50 mL of each filtrate into separate mixing cylinders.

6. Pipet 1.0 mL of the 2,4-Dinitrophenylhydrazine solution into each mixing cylinder. Stopper and shake gently. Wait exactly 20 minutes.

7. Pipet 1.0 mL of the 33% potassium hydroxide solution into each mixing cylinder. Then add 5 mL of pyridine stabilizer to each and stopper. Mix well and wait 2 minutes for colour stabilization.

8. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

9. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds

to the zero mark on the card.

10. Replace the Zero Scale card with the METHYL ETHYL KETONE in SOIL card.

11. Fill one cuvette to the 25 mL mark from the cylinder labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

12. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

13. Fill the other cuvette to the 25 mL mark from the cylinder labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the methyl ethyl ketone concentration from the card and record this result.

METHYL ETHYL KETONE IN WATER AND WASTEWATER

2,4-DINITROPHENYLHYDRAZINE METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Permagon Press, 1963.

Range: 0 - 400 mg/L

Common Synonyms: MEK, 2-Butanone, Ethyl Methyl Ketone

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - coughing, shortness of breath, headache, dizziness, irritation of respiratory tract. Ingestion- irritation of respiratory tract, nausea, vomiting. Contact- skin- defatting and dermatitis; eyes- irritation and burning.
Toxicology: Highly toxic by ingestion, moderately toxic by contact.
TLV - (inhalation) 200 ppm; 590 mg/cubic meter
Short-term Inhalation Limits - 300 ppm; 885 mg/cubic meter (15 min).

Environment:
Fish toxicity: 5640 ppm/48h/bluegill/TLm /freshwater
Aquatic Toxicity Rating - > 1000 ppm/96h/TLm/freshwater.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
5 x 10 mL Disposable Pipets
2 x 150 mL Beakers
1 x 100 mL Volumetric Flask
Timing Device

REQUIRED REAGENTS

Pyridine Stabilizer
33% Potassium Hydroxide Solution
2,4-Dinitrophenylhydrazine Solution

Preparation of Pyridine Stabilizer

Add 100 mL of deionized water to 400 mL of pyridine. Mix thouroughly. Store in a brown glass bottle.

Preparation of 33% Potassium Hydroxide Solution

Dissolve 33.0 g of potassium hydroxide in a 150 mL beaker containing 100 mL of methanol.

Preparation of 2,4-Dinitrophenylhydrazine solution

Dissolve 0.100 g of 2,4-Dinitrophenylhydrazine in a 100 mL beaker containing 50 mL of methanol and 4 mL of concentrated hydrochloric acid. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. From an uncontaminated source in the area obtain a 100 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 100 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Pipet 2.0 mL of the 2,4-Dinitrophenylhydrazine solution into each flask. Stopper and shake gently. Wait exactly 20 minutes.

4. Pipet 2.0 mL of the 33% potassium hydroxide solution into each flask. Then add 10 mL of pyridine stabilizer to each and mix well and wait 2-3 minutes for colour stabilization.

5. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

6. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

7. Replace the Zero Scale card with the METHYL ETHYL KETONE in WATER card.

8. Fill one cuvette to the 25 mL mark from the flask labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

9. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

10. Fill the other cuvette to the 25 mL mark from the flask labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the methyl ethyl ketone concentration from the card and record this result.

METHYL ISOBUTYL KETONE IN SOILS

2,4-DINITROPHENYLHYDRAZINE METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Permagon Press, 1963.

Range: 0 - 500 mg/kg

Common Synonyms: MIBK, Hexone, 4-Methyl-2-Pentanone, 2-Methyl-4-Pentanone, Isobutyl Methyl Ketone.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of respiratory tract, headache, nausea, dizziness, unconsciousness, CNS depressant. Ingestion - irritation of gastrointestinal tract, nausea, vomiting. Contact - skin- defatting and irritation; eyes- irritation and burning sensation.
Toxicology: Moderately toxic by all routes.
TLV - (inhalation) 50 ppm; 205 mg/cubic meter
Short-term Inhalation Limits - 75 ppm; 300 mg/cubic meter (15 min).

Environment:
Aquatic Toxicity Rating - > 1000 ppm/96h/TLm/freshwater.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
2 x 100 mL Mixing Cylinders
5 x 10 mL Disposable Pipets
2 x 150 mL Beakers
1 x 100 mL Volumetric Flask
Timing Device
Whatman #1 Filter Paper
2 x Long Stemmed Funnels
Balance

REQUIRED REAGENTS

Pyridine Stabilizer
33% Potassium Hydroxide Solution
2,4-Dinitrophenylhydrazine Solution

Preparation of Pyridine Stabilizer

Add 100 mL of deionized water to 400 mL of pyridine. Mix thouroughly. Store in a brown glass bottle.

Preparation of 33% Potassium Hydroxide Solution

Dissolve 33.0 g of potassium hydroxide in a 150 mL beaker containing 100 mL of methanol.

Preparation of 2,4-Dinitrophenylhydrazine solution

Dissolve 0.100 g of 2,4-Dinitrophenylhydrazine in a 100 mL beaker containing 50 mL of methanol and 4 mL of concentrated hydrochloric acid. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Transfer 50 mL of each filtrate into separate mixing cylinders.

6. Pipet 1.0 mL of the 2,4-Dinitrophenylhydrazine solution into each mixing cylinder. Stopper and shake gently. Wait exactly 20 minutes.

7. Pipet 1.0 mL of the 33% potassium hydroxide solution into each mixing cylinder. Then add 5 mL of pyridine stabilizer to each and stopper. Mix well and wait 2 minutes for colour stabilization.

8. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

9. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds

to the zero mark on the card.

10. Replace the Zero Scale card with the METHYL ISOBYTYL KETONE in SOIL card.

11. Fill one cuvette to the 25 mL mark from the cylinder labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

12. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

13. Fill the other cuvette to the 25 mL mark from the cylinder labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the methyl isobutyl ketone concentration from the card and record this result.

METHYL ISOBUTYL KETONE IN WATER AND WASTEWATER

2,4-DINITROPHENYLHYDRAZINE METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Permagon Press, 1963.

Range: 0 - 400 mg/L

Common Synonyms: MIBK, Hexone, 4-Methyl-2-Pentanone, 2-Methyl-4-Pentanone, Isobutyl Methyl Ketone.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of respiratory tract, headache, nausea, dizziness, unconsciousness, CNS depressant. Ingestion - irritation of gastrointestinal tract, nausea, vomiting. Contact - skin- defatting and irritation; eyes- irritation and burning sensation.
Toxicology: Moderately toxic by all routes.
TLV - (inhalation) 50 ppm; 205 mg/cubic meter
Short-term Inhalation Limits - 75 ppm; 300 mg/cubic meter (15 min).

Environment:
Aquatic Toxicity Rating - > 1000 ppm/96h/TLm/freshwater.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
5 x 10 mL Disposable Pipets
2 x 150 mL Beakers
1 x 100 mL Volumetric Flask
Timing Device

REQUIRED REAGENTS

Pyridine Stabilizer
33% Potassium Hydroxide Solution
2,4-Dinitrophenylhydrazine Solution

Preparation of Pyridine Stabilizer -

Add 100 mL of deionized water to 400 mL of pyridine. Mix

thouroughly. Store in a brown glass bottle.

Preparation of 33% Potassium Hydroxide Solution -

Dissolve 33.0 g of potassium hydroxide in a 150 mL beaker containing 100 mL of methanol.

Preparation of 2,4-Dinitrophenylhydrazine solution -

Dissolve 0.100 g of 2,4-Dinitrophenylhydrazine in a 100 mL beaker containing 50 mL of methanol and 4 mL of concentrated hydrochloric acid. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. From an uncontaminated source in the area obtain a 100 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 100 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Pipet 2.0 mL of the 2,4-Dinitrophenylhydrazine solution into each flask. Stopper and shake gently. Wait exactly 20 minutes.

4. Pipet 2.0 mL of the 33% potassium hydroxide solution into each flask. Then add 10 mL of pyridine stabilizer to each and mix well and wait 2-3 minutes for colour stabilization.

5. Adjust the Wavelength Dial to read 480 nm.

6. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

7. Replace the Zero Scale card with the METHYL ISOBUTYL KETONE in WATER card.

8. Fill one cuvette to the 25 mL mark from the flask labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

9. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

10. Fill the other cuvette to the 25 mL mark from the flask labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the methyl isobutyl ketone concentration from the card and record this result.

MORPHOLINE IN SOIL

PERCHLORIC ACID TITRIMETRIC METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Pergamon Press, New York, 1963.

Range: 500 - 5000 mg/kg

Common Synonyms: Tetrahydro-2H-1,4-oxazine, Tetrahydro-1,4-oxazine, Diethylene Oximide, Diethylene Imidoxide.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - corneal edema, foggy vision and irritation to respiratory tract. Ingestion - irritation to intestinal tract. Contact - severe irritation on contact with skin and eyes.
Toxicology: Highly toxic by contact, moderately by ingestion.
TLV = 20ppm

Environment:
Aquatic Toxicity Rating- 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 250 mL Erlenmeyer flasks
- 2 x magnetic stirring bars
- 50 mL graduated cylinder
- 10 mL graduated cylinder
- 25 mL buret
- Magnetic stirrer
- 2 x funnels
- filter paper
- 2 x 250 mL beaker

REQUIRED REAGENTS

- Methanol
- Acrylonitrile
- Methyl Cellosolve
- Acetic Anhydride
- 0.1N Perchloric acid
- Thymol Blue / Xylene Cyanol FF indicator solution

Preparation of Perchloric acid solution, 0.1N -

Measure 10.7 mL of 70% perchloric acid and transfer to a 250 mL volumetric flask. Dilute to the mark with Methyl Cellosolve. Transfer 50 mL from this to another 250 mL volumetric flask. Dilute to the mark with Methyl Cellosolve. Use this to titrate.

Preparation of Thymol Blue/Xylene Cyanol FF indicator solution -

Dissolve 0.3 grams of thymol blue and 0.08 grams of xylene cyanol FF in 100 mL of dimethylformamide (DMF).

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 250 mL beakers. Label the beakers to match the originating flask.

5. Put a magnetic stirring bar in each beaker.

6. Add 5 mL of Acrylonitrile, using a clean 10 mL graduated cylinder, to each beaker. Then add 10 mL of Methanol to each beaker, shake well to mix and wait 5 minutes for reaction to proceed.

5. Add 50 mL of Methyl Cellosolve to each beaker and stir. Slowly add 20 mL of Acetic Anhydride to each beaker. Caution- reaction is endothermic and the beakers may get hot!

6. Allow beakers to cool for 1 minute, then add 1 mL of thymol blue / xylene cyanol FF indicator solution to each beaker.

7. Fill the buret with 0.5N Perchloric acid. Record the initial buret reading. Place the BLANK sample on the magnetic stirrer. Turn stirrer to low speed. Titrate the BLANK sample with Perchloric acid until the green colour has disappeared. Record final buret reading.

8. Refill the buret. Record initial buret reading. Titrate the TEST sample as above. Record final buret reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Morpholinee by solving the following equation:

$$\text{Concentration (mg/kg)} = [\ (\text{adjusted titrant volume}) \times (\text{titrant concentration}) \times 10800\ ]$$

where titrant concentration is 0.1 unless above Perchloric acid solution is not used.

MORPHOLINE IN WATER

PERCHLORIC ACID TITRIMETRIC METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Pergamon Press, New York, 1963.

Range: 100 - 2000 mg/L

Common Synonyms: Tetrahydro-2H-1,4-oxazine, Tetrahydro-1,4-oxazine, Diethylene Oximide, Diethylene Imidoxide.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - corneal edema, foggy vision and irritation to respiratory tracts. Ingestion - irritation to intestinal tract. Contact - severe irritation on contact with skin and eyes.
Toxicology: Highly toxic by contact, moderately by ingestion.
TLV = 20ppm

Environment:
Aquatic Toxicity Rating- 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer flasks
2 x magnetic stirring bars
50 mL graduated cylinder
10 mL graduated cylinder
25 mL buret
Magnetic stirrer

REQUIRED REAGENTS

Methanol
Acryonitrile
Methyl Cellosolve
Acetic Anhydride
0.5N Perchloric acid
Thymol Blue / Xylene Cyanol FF indicator solution

Preparation of Perchloric acid solution, 0.5N -

Measure 10.7 mL of 70% perchloric acid and transfer to a 250 mL volumetric flask. Dilute to the mark with Methyl Cellosolve.

Preparation of Thymol Blue/Xylene Cyanol FF indicator solution -

Dissolve 0.3 grams of thymol blue and 0.08 grams of xylene cyanol FF in 100 mL of dimethylformamide (DMF).

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Put a magnetic stirring bar in each flask.

4. Add 5 mL of Acrylonitrile, using a clean 10 mL graduated cylinder, to each flask. Then add 10 mL of Methanol to each flask, shake well to mix and wait 5 minutes for reaction to proceed.

5. Add 50 mL of Methyl Cellosolve to each flask and stir. Slowly add 20 mL of Acetic Anhydride to each flask. Caution - reaction is endothermic and the flasks may get hot!

6. Allow flasks to cool for 1 minute, then add 1 mL of thymol blue / xylene cyanol FF indicator solution to each flask.

7. Fill the buret with 0.5N Perchloric acid. Record the initial buret reading. Place the BLANK sample on the magnetic stirrer. Turn stirrer to low speed. Titrate the BLANK sample with Perchloric acid until the green colour has disappeared. Record final buret reading.

8. Refill the buret. Record initial buret reading. Titrate the TEST sample as above. Record final buret reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Morpholine by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x (titrant concentration) x 1675]

where titrant concentration is 0.5 unless standard Perchloric acid solution is not used.

NITRIC ACID IN WATER

SODIUM HYDROXIDE TITRATION METHOD

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0 - 10000 mg/L

Common Synonyms: Red Fuming Nitric Acid, White Fuming Nitric Acid.

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin- yellow discolouration, burning, inflammation; eyes- burning, watering, ulceration, loss of sight. Inhalation - irritation of mucous membranes, difficulty breathing, coughing fits, nausea, muscular weakness. Ingestion- irritation and burning, intense thirst, cramps, nausea, vomiting, convulsions, coma.
Toxicology: Highly toxic to skin, eyes, and mucous membranes.
TLV - 2 ppm; 5 mg/cubic meter

Environment:
Aquatic Toxicity Rating - 10 to 100 ppm/96h/TLm/freshwater.

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 50 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
1 x 25 mL Burette

REQUIRED REAGENTS

Phenolphthalein indicator solution
0.02N Sodium Hydroxide solution
*Note: All required reagents supplied by Hach company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Fill the 0 - 25 mL burette with 0.02N Sodium Hydroxide solution and record the initial burette reading.

4. Add 6 drops of phenolphthalein solution to each flask.

5. Titrate the BLANK water sample against the sodium hydroxide solution; add slowly, with continuous swirling until a pink colour appears for more than 30 seconds.

6. Record the final burette reading.

7. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Nitric Acid by solving the following equation:

$$\text{Concentration (mg/L)} = [\ (\text{adjusted titrant volume}) \times 35.4\ ]$$

NITRILOTRIACETIC ACID IN SOIL

HYDROLYSIS AND SODIUM HYDROXIDE TITRATION

Reference: Smith, D.M., Bryant, W.M.D., J. Am. Chem. Soc., 58, 2452, 1936.

Range: 250 - 5000 mg/kg

Common Synonyms: N,N-Bis(carboxymethyl)glycine, Triglycollamic Acid, Triglycine, NTA, Aminotriacetic Acid.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation and Ingestion - no data. Contact-irritation of skin and eyes.
Toxicology: no data.

Environment:
Aquatic Toxicity Rating - 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 125 Erlenmeyer flasks
- 2 x funnel
- filter paper
- 2 x 150 mL beaker
- 50 mL graduated cylinder
- 2 x magnetic stirring bars
- magnetic stirrer
- 25 mL buret

REQUIRED REAGENTS

- 1:1 Pyridine/Water solution
- 0.02N Sodium Hydroxide
- Thymol blue indicator solution

Preparation of 1:1 pyridine/water solution -

Combine 75 mL of deionized water with 75 mL of pyridine in a 500 mL bottle with a screw-top lid.

Preparation of Thymol blue indicator solution -

Dissolve 0.4 grams of Thymol blue in 100 mL of deionized water and store in a 150 mL plastic bottle with lid.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of pyridine/water solution to each flask. Mix well for 5 minutes.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Put a magnetic stirring bar in each beaker. Add 6 drops of Thymol blue indicator to each sample.

6. Fill the buret with 0.02N Sodium Hydroxide solution. Record the initial buret reading. Place the BLANK sample on the magnetic stirrer and turn stirrer on to low speed. Titrate the BLANK sample with Sodium Hydroxide until the solution changes colour from yellow to blue-green. Record the final buret reading.

7. Refill the buret. Record initial buret reading. Titrate the TEST sample as above. Record final buret reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Nitrilotriacetic acid by solving the following equation:

$$\text{Concentration (mg/kg)} = [\ (\text{adjusted titrant volume}) \times 295\ ]$$

NITRILOTRIACETIC ACID IN WATER

SODIUM HYDROXIDE TITRATION

Reference: Smith, D.M., Bryant, W.M.D., J. Am. Chem. Soc., 58, 2452, 1936.

Range: 40 - 1500 mg/L

Common Synonyms: N,N-Bis(carboxymethyl)glycine, triglycollamic acid, triglycine, NTA, Aminotriacetic acid.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation and Ingestion - no data. Contact-irritation of skin and eyes.
Toxicology: no data.

Environment:
Aquatic Toxicity Rating - 100 to 1000/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
2 x 250 mL Erlenmeyer flasks
50 mL graduated cylinder
2 x magnetic stirring bars
magnetic stirrer
25 mL buret

REQUIRED REAGENTS

0.02N Sodium Hydroxide
Phenolphthalein indicator solution

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Put a magnetic stirring bar in each flask. Add 6 drops of Phenolphthalein indicator to each sample.

4. Fill the burette with 0.02N Sodium Hydroxide solution. Record the initial burette reading. Place the BLANK sample on the magnetic stirrer and turn stirrer on to low speed. Titrate the BLANK sample with Sodium Hydroxide until pink colour remains for at least 1 minute. Record the final burette reading.

5. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Nitrilotriacetic acid by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 36.7]

PHOSPHORIC ACID IN WATER AND WASTEWATER

AMINO ACID COLORIMETRIC TEST

Reference: APHA Standard Methods, 12 Ed.,1965.

Range: 0-15 mg/L

Common Synonyms: Orthophosphoric Acid

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of mucous membranes, watering of eyes, difficulty breathing, salivation, nausea. Ingestion- pain in swallowing, intense thirst, abdominal pain, nausea. Contact - skin- burning, inflammation, blistering; eyes- burning, watering.
Toxicology: Moderately toxic by ingestion and contact.
TLV - (inhalation) 1 mg/cubic meter.

Environment:
Aquatic Toxicity Rating - 100 to 1000 ppm/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 25 mL Graduated Cylinders
5 x 1 mL Disposable Pipets
Timing Device
2 x 250 mL Erlenmeyer Flasks

REQUIRED REAGENTS

Molybdate Reagent
Amino Acid pillows
*Note: all required reagents supplied by Hach Company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Pipet 1.0 mL of Molybdate Reagent into each flask.

4. Add 1 Amino Acid pillow to each flask and mix thouroughly. Wait 10 minutes for colour stabilization.

5. Adjust the Wavelength Dial of the spectrophotometer to read 530 nm.

5. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

6. Replace the Zero Scale card with the PHOSPHORIC ACID in WATER card.

7. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

8. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

9. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Phosphoric Acid concentration from the card and record this result.

PHOSPHORIC ACID IN WATER AND WASTEWATER

SODIUM HYDROXIDE TITRIMETRIC TEST

Reference: APHA Standard Methods, 12 Ed.,1965.

Range: 0- 10000 mg/L

Common Synonyms: Orthophosphoric Acid

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of mucous membranes, watering of eyes, difficulty breathing, salivation, nausea. Ingestion- pain in swallowing, intense thirst, abdominal pain, nausea. Contact - skin- burning, inflammation, blistering; eyes- burning, watering.
Toxicology: Moderately toxic by ingestion and contact.
TLV - (inhalation) 1 mg/cubic meter.

Environment:
Aquatic Toxicity Rating - 100 to 1000 ppm/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 50 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
1 x 25 mL Burette

REQUIRED REAGENTS

Phenolphthalein indicator solution
0.02N Sodium Hydroxide solution
*Note: All required reagents supplied by Hach company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by

filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Fill the 0 - 25 mL burette with 0.02N Sodium Hydroxide solution and record the initial burette reading.

4. Add 6 drops of phenolphthalein solution to each flask.

5. Titrate the BLANK water sample against the sodium hydroxide solution; add slowly, with continuous swirling until a pink colour appears for more than 30 seconds.

6. Record the final burette reading.

7. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Phosphoric Acid by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 20]

PHTHALIC ANHYDRIDE IN SOIL

SODIUM HYDROXIDE TITRATION

Reference: Smith, D.M., Bryant, W.M.D., J. Am. Chem. Soc., 58, 2452, 1936.

Range: 200 - 4000 mg/kg

Common Synonyms: 1,3-Isobenzofurandione, PAN, Phthalandione

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - sore throat, coughing, respiratory difficulty, cyanosis. Ingestion - burning sensation, cramps, nausea, dizziness, diarrhea, shock, convulsions, coma. Contact-skin - irritation, inflammation, brown staining ; eyes-stinging, irritation.
Toxicology: Moderately toxic by all routes.

Environment:
Aquatic Toxicity Rating - 10 to 100/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 125 Erlenmeyer flasks
- 2 x funnel
- filter paper
- 2 x 150 mL beaker
- 50 mL graduated cylinder
- 2 x magnetic stirring bars
- magnetic stirrer
- 25 mL buret

REQUIRED REAGENTS

- Methanol
- 0.02N Sodium Hydroxide
- Thymol blue indicator solution

Preparation of Thymol blue indicator solution -

Dissolve 0.4 grams of Thymol blue in 100 mL of deionized water and store in a 150 mL plastic bottle with lid.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of methanol to each flask. Mix well for 15 minutes.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Put a magnetic stirring bar in each beaker. Add 6 drops of Thymol blue indicator to each sample.

6. Fill the burette with 0.02N Sodium Hydroxide solution. Record the initial burette reading. Place the BLANK sample on the magnetic stirrer and turn stirrer on to low speed. Titrate the BLANK sample with Sodium Hydroxide until the solution changes colour from yellow to blue-green. Record the final burette reading.

7. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Phthalic Anhydride by solving the following equation:

$$\text{Concentration (mg/kg)} = [\ (\text{adjusted titrant volume}) \times 355\ ]$$

POTASSIUM CHLORIDE IN SOILS

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/kg

Common Synonyms: Potash, Potassium Muriate, Muriate of Potash.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - slight irritation of nose, sneezing. Ingestion - nausea, vomiting. Contact - eyes- irritation and inflammation; skin- irritation.
Toxicology: Low toxicity by all routes.

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 2 x Long-Stem Funnels
- Whatman 12.5 cm #1 Filter Paper
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 1 x 100 mL Volumetric Flask
- 2 x 50 mL Beakers
- 4 x 150 mL Beakers
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer
- pH Meter and Electrode

REQUIRED REAGENTS

- 0.1 N Nitric Acid solution
- 0.014 N Mercuric Nitrate solution
- Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and

label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Pipet 25 mL of each filtrate into two more separate, clean 150 mL beakers. Add 75 mL of deionized water to each using the graduated cylinder.

6. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

7. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

8. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

9. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

10. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Potassium Chloride by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x 421]

POTASSIUM CHLORIDE IN WATER AND WASTEWATER

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/L

Common Synonyms: Potash, Potassium Muriate, Muriate of Potash.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - slight irritation of nose, sneezing. Ingestion - nausea, vomiting. Contact - eyes- irritation and inflammation; skin- irritation.
Toxicology: Low toxicity by all routes.

EXPERIMENTAL REQUIREMENTS

Apparatus:
1 x 100 mL Graduated Cylinder
2 x 250 mL Erlenmeyer Flasks
2 x Long-Stem Funnels
Whatman 12.5 cm #1 Filter Paper
1 x 25 mL Burette
1 x 1000 mL Volumetric Flask
1 x 100 mL Volumetric Flask
2 x 50 mL Beakers
4 x 150 mL Beakers
Magnetic Stir Bars
Battery Operated Magnetic Stirrer
pH Meter and Electrode

REQUIRED REAGENTS

0.1 N Nitric Acid solution
0.014 N Mercuric Nitrate solution
Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and

label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thouroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Pipet 25 mL of each filtrate into two more separate, clean 150 mL beakers. Add 75 mL of deionized water to each using the graduated cylinder.

6. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

7. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

8. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

9. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

10. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Potassim Chloride by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 21.03]

POTASSIUM HYDROXIDE IN WATER AND WASTEWATER

SULFURIC ACID TITRATION

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0-10000 mg/L

Common Synonyms: Lye, Caustic Potash, Potassium Hydrate, Potassa.

HAZARDOUS DATA

Human Health
Symptoms: Contact with solutions can cause severe burns. Contact - skin- burns; eyes- burns, severe damage. Inhalation- (dust or mist) may damage upper respiratory tract or even lung tissue. Ingestion - can result in severe damage to mucous membranes, nausea, vomiting and diarrhea.
Toxicology: Highly toxic by ingestion and inhalation.
TLV - (inhalation) 2 mg/cubic meter

Environment:
Aquatic Toxicity Rating - 10 to 100 ppm/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer Flasks
2 x 50 mL Graduated Cylinders
1 x 25 mL Burette
Magnetic Stir Bars
Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

0.02 N Sulfuric Acid
Phenolphthalein indicator solution
Bromocresol Green - Methyl Red indicator solution
*Note: All required reagents are supplied by the Hach Company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean

250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Fill the burette with 0.02 N Sulfuric Acid and record the initial burette reading.

4. Put a magnetic stir bar in the BLANK flask, put the flask on the stirrer and turn ON. Add 6 drops of the phenolphthalein indicator to the BLANK solution and mix. A pink colour should develop; if the solution remains colourless, proceed to step 6.

5. Titrate the BLANK sample by slowly adding the 0.02 N sulfuric acid from the burette. Stop the titration when the water sample becomes colourless.

6. Add 6 drops of the bromocresol green - methyl red indicator to the BLANK solution. A blue-green colour will appear upon addition of the indicator.

7. Continue (or start) titrating with 0.02 N sulfuric acid until the solution turns a pink-orange colour. Record the final burette reading.

8. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Potassium Hydroxide by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 26.2]

POTASSIUM SULFATE IN SOILS

THORIN TITRIMETRIC TEST

Reference: Environment Canada, Analytical Methods Manual, 1974.

Range: 0-25000 mg/kg

Common Synonyms:

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of nose and throat. Ingestion - stomach pains. Contact - skin and eyes - irritated by dust.
Toxicology: Low toxicity by all routes.

EXPERIMENTAL REQUIREMENTS

Apparatus: 1 x 100 mL Graduated Cylinder
2 x 250 mL Erlenmeyer Flasks
1 x 125 mL Erlenmeyer Flask fitted with a bored #6 Rubber Stopper
2 x long-stem funnels
Whatman 12.5 cm #1 Filter Paper
1 x 25 mL Burette
1 x 1000 mL Volumetric Flask
5 x 10 mL Disposable Pipets
6 x 150 mL Beakers
125 mL Separatory Funnel
Magnetic Stir Bars
Battery Operated Magnetic Stirrer
Glass Wool
Handpump
pH Meter & Electrode

REQUIRED REAGENTS

20% Hydrochloric Acid solution
1% Hydrochloric Acid solution
1% Ammonium Hydroxide solution
0.00164 N Barium Chloride solution
Ion - Exchange Resin (Dowex 50W-X8)
Thorin indicator solution
Ethanol

Preparation of 20% Hydrochloric Acid solution -

To a 400 mL beaker containing 200 mL of deionized water, slowly add 50 mL of concentrated hydrochloric acid.

Preparation of 1% Hydrochloric Acid -

To a 100 mL beaker containing 95 mL of deionized water, slowly add 5 mL of the 20% hydrochloric acid solution.

Preparation of 1% Ammonium Hydroxide solution -

To a 100 mL beaker containing 99 mL of deionized water, add 1 mL of concentrated ammonium hydroxide.

Preparation of 0.00164 N Barium Chloride solution -

Dissolve 0.400 g of Barium Chloride in a 500 mL beaker containing 400 mL of deionized water. Adjust the solution to pH 3.9 by adding 1% hydrochloric acid solution dropwise. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water.

Preparation of Ion-Exchange Resin -

Insert a small glass wool plug into the neck of a 125 mL separatory funnel. Add 25 g of Dowex 50W-X8 cation exchange resin.

Preparation of Thorin indicator -

Dissolve 0.2 g of Thorin in deionized water in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Prepare the ion-exchange column (as illustrated) by passing 25 mL of 20% hydrochloric acid through it. Discard the spent acid.

6. Pass 2 x 20 mL aliquots of deionized water through the ion-exchange column. Discard the spent water.

7. Pass 20 mL of the BLANK solution through the ion-exchange column. Discard the spent solution.

8. Pass a 30 mL aliquot of the BLANK solution through the ion-exchange column. Pipet 10 mL of this into a clean 150 mL beaker.

9. Repeat steps 5 through 8 using the TEST sample in place of the BLANK sample.

10. Using the graduated cylinder, add 40 mL of Ethanol to each beaker.

11. Adjust both solutions to pH 3.8-4.0 using 1% hydrochloric acid or 1% ammonium hydroxide as required.

12. Add 2-3 drops of thorin indicator to each beaker. The solution should now be orange in colour.

13. Fill the 0-25 mL burette with the barium chloride solution. Record the initial burette reading.

14. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

15. Titrate the BLANK solution by slowly adding the barium chloride until the first permanent pink colour appears. Record the final volume.

16. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Potassium Sulfate by solving the following equation:

$$\text{Concentration (mg/kg)} = [\ (\text{adjusted titrant volume}) \times 285\ ]$$

PROPYLENE GLYCOL IN SOIL

PERIODIC ACID TEST METHOD

Reference: Lehman, A.J., Newman, H.W., J. Pharmacol. Exptl. Therap., 60, 312, 1937.

Range: 150 - 3000 mg/kg

Common Synonyms: Methylene Glycol, Monopropylene Glycol, Methyl Glycol, Trimethyl Glycol.

HAZARDOUS DATA

Human Health:
Symptoms: Inhalation: intoxication, nose and eye irritation, headache, mental confusion, nausea and vomiting. Ingestion: gastrointestinal irritation and symptoms similar to above.
Contact: Skin- may be absorbed ; Eyes- redness and pain
Toxicology: Low toxicity by all routes

Environment:
Aquatic Toxicity Rating: >1000 ppm/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 250 mL Erlenmeyer flasks
- 25 mL burette
- Battery operated magnetic stirrer
- 2 x magnetic stirring bars
- 50 mL graduated cylinder
- 1 x 100 mL volumetric flask
- 1 x 500 mL volumetric flask
- 2 x 1000 mL volumetric flask
- 2 x 50 mL beakers

REQUIRED REAGENTS

- 0.5 M Periodic Acid
- 0.1 N Arsenious Acid
- 0.1 N Iodine
- 15% Potassium Iodide solution
- Saturated Sodium Bicarbonate solution
- Starch Indicator solution
- Arsenic trioxide (for standardization of Iodine)

Preparation of Periodic Acid solution, 0.5 M

Dissolve 11.397 grams of periodic acid in 50 mL of deionized water in a 150 mL beaker. Add 6.5 mL of concentrated sulfuric acid to the beaker. Transfer the solution to a 500 mL volumetric flask and dilute to the 500 mL mark with deionized water.

Preparation of Saturated Sodium Bicarbonate solution -

Add 20 g of sodium bicarbonate to a 250 mL beaker containing 100 mL of deionized water. NOTE - not all bicarbonate will dissolve.

Preparation of 15% Potassium Iodide -

Dissolve 15.000 g of potassium iodide in 50 mL of deionized water in a 100 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

Preparation of Starch solution -

Dissolve 1.0 g of soluble starch in 500 mL of hot (preferably boiling) deionized water.

Preparation of 0.1 N Iodine solution -

Dissolve 12.69 g of iodine in a 250 mL beaker containing 40 g of potassium iodide and 25 mL of deionized water. Stir to dissolve the iodine. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water. Mix the solution thoroughly.

Preparation of 0.1 N Arsenious Acid solution -

Dissolve 5.000 g of Sodium Arsenite in a 50 mL beaker containing 3.0 g of sodium hydroxide and 10 mL of deionized water. Allow the solution to stand, swirling it occasionally until the solid has completely dissolved. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water. Mix the solution thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Put a magnetic stir bar in each of the two flasks.

4. Add 5.0 mL of 0.1M Periodic Acid to each flask. Put one of the flasks on the magnetic stirrer. After 10 minutes, switch the flasks and stir the other flask for 10 minutes for a total reaction time of 20 minutes.

6. Add 10 mL of saturated Sodium Bicarbonate to each flask. Then add 0.5 mL of Potassium Iodide solution to each flask.

7. Add 20 mL of Arsenious Acid solution to each sample.

8. Add approximately 3 grams of Sodium Bicarbonate to each flask and stir well.

9. Fill the burette with 0.1N Iodine and record burette reading. Add 5 mL of Starch indicator solution to the BLANK sample and titrate with 0.1N Iodine until the blue colour remains stable (1 minute). Record final burette reading.

10. Refill burette and record burette reading. Add 5 mL of Starch indicator solution to the TEST sample and titrate until the blue colour remains stable (1 minute). Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Propylene Glycol by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x

(titrant concentration) x 3420]

where titrant concentration is 0.1 unless standard Iodine solution is not used.

PROPYLENE GLYCOL IN WATER AND WASTEWATER

PERIODIC ACID TEST METHOD

Reference: Lehman, A.J., Newman, H.W., J. Pharmacol. Exptl. Therap., 60, 312, 1937.

Range: 0 - 500 mg/L

Common Synonyms: Methylene Glycol, Monopropylene Glycol, Methyl Glycol, Trimethyl Glycol.

HAZARDOUS DATA

Human Health:
Symptoms: Inhalation: intoxication, nose and eye irritation, headache, mental confusion, nausea and vomiting. Ingestion: gastrointestinal irritation and symptoms similar to above.
Contact: Skin- may be absorbed ; Eyes- redness and pain
Toxicology: Low toxicity by all routes

Environment:
Aquatic Toxicity Rating: >1000 ppm/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 250 mL Erlenmeyer flasks
- 25 mL buret
- Battery operated magnetic stirrer
- 2 x magnetic stirring bars
- 50 mL graduated cylinder
- 1 x 100 mL volumetric flask
- 1 x 500 mL volumetric flask
- 2 x 1000 mL volumetric flask
- 2 x 50 mL beakers

REQUIRED REAGENTS

- 0.5 M Periodic Acid
- 0.1 N Arsenious Acid
- 0.1 N Iodine
- 15% Potassium Iodide solution
- Saturated Sodium Bicarbonate solution
- Starch Indicator solution
- Arsenic trioxide (for standardization of Iodine)

Preparation of Periodic Acid solution, 0.5 M

Dissolve 11.397 grams of periodic acid in 50 mL of deionized water in a 150 mL beaker. Add 6.5 mL of concentrated sulfuric acid to the beaker. Transfer the solution to a 500 mL volumetric flask and dilute to the 500 mL mark with deionized water.

Preparation of Saturated Sodium Bicarbonate solution -

Add 20 g of sodium bicarbonate to a 250 mL beaker containing 100 mL of deionized water. NOTE - not all bicarbonate will dissolve.

Preparation of 15% Potassium Iodide -

Dissolve 15.000 g of potassium iodide in 50 mL of deionized water in a 100 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

Preparation of Starch solution -

Dissolve 1.0 g of soluble starch in 500 mL of hot (preferably boiling) deionized water.

Preparation of 0.1 N Iodine solution -

Dissolve 12.69 g of iodine in a 250 mL beaker containing 40 g of potassium iodide and 25 mL of deionized water. Stir to dissolve the iodine. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water. Mix the solution thoroughly.

Preparation of 0.1 N Arsenious Acid solution -

Dissolve 5.000 g of Sodium Arsenite in a 50 mL beaker containing 3.0 g of sodium hydroxide and 10 mL of deionized water. Allow the solution to stand, swirling it occasionally until the solid has completely dissolved. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water. Mix the solution thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Put a magnetic stir bar in each of the two flasks.

4. Add 5.0 mL of 0.5 M Periodic Acid to each flask. Put one of the flasks on the magnetic stirrer. After 10 minutes, switch the flasks and stir the other flask for 10 minutes for a total reaction time of 20 minutes.

5. Add 10 mL of saturated Sodium Bicarbonate to each flask. Then add 0.5 mL of Potassium Iodide solution to each flask.

6. Add 20 mL of Arsenious Acid solution to each sample.

7. Add approximately 3 grams of Sodium Bicarbonate to each flask and stir well.

8. Fill the burette with 0.1N Iodine and record burette reading. Add 5 mL of Starch indicator solution to the BLANK sample and titrate with 0.1N Iodine until the blue colour remains stable (1 minute). Record final burette reading.

9. Refill buret and record buret reading. Add 5 mL of Starch indicator solution to the TEST sample and titrate until the blue colour remains stable (1 minute). Record final buret reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial buret reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Propylene Glycol by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x (titrant concentration) x 75.6]

where titrant concentration is 0.1 unless standard Iodine solution is not used.

SODIUM CARBONATE IN WATER AND WASTEWATER

SULFURIC ACID TITRATION

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0-10000 mg/L

Common Synonyms: Calcined Soda, Soda Ash, Soda Nonohydrate, Crystal Carbonate, Carbonic Acid Disodium Salt.

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin and eyes- irritation from dust. Inhalation - irritation of respiratory tract, difficulty breathing, coughing, sneezing. Ingestion - of large amounts is corrosive to the gastrointestinal tract causing cramps, vomiting, diarrhea, and possible circulatory collapse.
Toxicology: Moderately toxic by ingestion, low toxicity otherwise.

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 2 x 250 mL Erlenmeyer Flasks
- 2 x 50 mL Graduated Cylinders
- 1 x 25 mL Burette
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

- 0.02 N Sulfuric Acid
- Phenolphthalein indicator solution
- Bromocresol Green - Methyl Red indicator solution

*Note: All required reagents are supplied by the Hach Company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Fill the burette with 0.02 N Sulfuric Acid and record the initial burette reading.

4. Put a magnetic stir bar in the BLANK flask, put the flask on the stirrer and turn ON. Add 6 drops of the phenolphthalein indicator to the BLANK solution and mix. A pink colour should develop; if the solution remains colourless, proceed to step 6.

5. Titrate the BLANK sample by slowly adding the 0.02 N sulfuric acid from the burette. Stop the titration when the water sample becomes colourless.

6. Add 6 drops of the bromocresol green - methyl red indicator to the BLANK solution. A blue-green colour will appear upon addition of the indicator.

7. Continue (or start) titrating with 0.02 N sulfuric acid until the solution turns a pink-orange colour. Record the final burette reading.

8. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Sodium Carbonate by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 24]

SODIUM CHLORIDE IN SOILS

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/kg

Common Synonyms: Halite, Rock Salt, Sea Salt, Common Salt.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - slight irritation of nose, sneezing. Ingestion - disagreeable taste, nausea, vomiting. Contact- eyes- irritation, watering; skin- irritation and inflammation.
Toxicology: Low toxicity by all routes.

Environment:
Land-Air: Salt concentrations of 1.3 - 2.6 g/L may affect sensitive crops. Concentrations of 2.6 - 5.0 g/L restricts yields of many crops. Greater than 5.1 g/L affects yields of most crops.

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 2 x long-stem funnels
- Whatman 12.5 cm #1 Filter Paper
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 1 x 100 mL Volumetric Flask
- 2 x 50 mL Beakers
- 4 x 150 mL Beakers
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer
- pH Meter and Electrode

REQUIRED REAGENTS

- 0.1 N Nitric Acid solution
- 0.014 N Mercuric Nitrate solution
- Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Pipet 25 mL of each filtrate into two more separate, clean 150 mL beakers. Add 75 mL of deionized water to each using the graduated cylinder.

6. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

7. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

8. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

9. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

10. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Sodium Chloride by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x 329]

SODIUM CHLORIDE IN WATER AND WASTEWATER

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/L

Common Synonyms: Halite, Rock Salt, Sea Salt, Common Salt.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - slight irritation of nose, sneezing. Ingestion - disagreeable taste, nausea, vomiting. Contact- eyes- irritation, watering; skin- irritation and inflammation.
Toxicology: Low toxicity by all routes.

Environment:
Land-Air: Salt concentrations of 1.3 - 2.6 g/L may affect sensitive crops. Concentrations of 2.6 - 5.0 g/L restricts yields of many crops. Greater than 5.1 g/L affects yields of most crops.

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 2 x long-stem funnels
- Whatman 12.5 cm #1 Filter Paper
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 1 x 100 mL Volumetric Flask
- 2 x 50 mL Beakers
- 4 x 150 mL Beakers
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer
- pH Meter and Electrode

REQUIREL REAGENTS

- 0.1 N Nitric Acid solution
- 0.014 N Mercuric Nitrate solution
- Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Pipet 25 mL of each filtrate into two more separate, clean 150 mL beakers. Add 75 mL of deionized water to each using the graduated cylinder.

6. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

7. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

8. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

9. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

10. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Sodium Chloride by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 16.48]

SODIUM HYDROXIDE IN WATER AND WASTEWATER

SULFURIC ACID TITRATION

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0-10000 mg/L

Common Synonyms: Caustic Soda, Caustic Lye, Soda Lye, Sodium Hydrate, White Caustic.

HAZARDOUS DATA

Human Health
Symptoms: Contact - skin- severe burns, often resulting in deep ulcerations and scarring; eyes- solid or liquid causes severe damage. Inhalation - of dust or mist may cause damage to upper respiratory tract and even lung damage. Ingestion - severe damage to mucous membranes or deeper tissues. Penetration into vital areas may be fatal.
Toxicology: Highly toxic by all routes.
TLV - (inhalation) 2 mg/cubic meter

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer Flasks
2 x 50 mL Graduated Cylinders
1 x 25 mL Burette
Magnetic Stir Bars
Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

0.02 N Sulfuric Acid
Phenolphthalein indicator solution
Bromocresol Green - Methyl Red indicator solution
*Note: All required reagents are supplied by the Hach Company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Fill the burette with 0.02 N Sulfuric Acid and record the initial burette reading.

4. Put a magnetic stir bar in the BLANK flask, put the flask on the stirrer and turn ON. Add 6 drops of the phenolphthalein indicator to the BLANK solution and mix. A pink colour should develop; if the solution remains colourless, proceed to step 6.

5. Titrate the BLANK sample by slowly adding the 0.02 N sulfuric acid from the burette. Stop the titration when the water sample becomes colourless.

6. Add 6 drops of the bromocresol green - methyl red indicator to the BLANK solution. A blue-green colour will appear upon addition of the indicator.

7. Continue (or start) titrating with 0.02 N sulfuric acid until the solution turns a pink-orange colour. Record the final burette reading.

8. Repeat steps 3 through 7 using the TEST sample, being sure to record both final and initial burette readings.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Sodium Hydroxide by solving the following equation:

$$\text{Concentration (mg/L)} = [\ (\text{adjusted titrant volume}) \times 33.3\ ]$$

SODIUM PHOSPHATES IN WATER AND WASTEWATER

AMINO ACID COLORIMETRIC TEST

Reference: APHA Standard Methods, 12 Ed.,1965.

Range: 0-70 mg/L

Common Synonyms: DSP, Phosphate of soda, Disodium Hydrogen Phosphate, Disodium Phosphate, Disodium Monohydrogen Phosphate.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - of dust may irritate nose and throat. Ingestion - injures mouth, throat, and gastrointestinal tract resulting in vomiting, cramps, and diarrhea, pain and burning. Contact - eyes- local irritation to chronic damage; skin- dermatitis.
Toxicology: Low toxicity by ingestion.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 25 mL Graduated Cylinders
5 x 1 mL Disposable Pipets
Timing Device
2 x 250 mL Erlenmeyer Flasks

REQUIRED REAGENTS

Molybdate Reagent
Amino Acid pillows
*Note: all required reagents supplied by Hach Company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the

flask TEST.

3. Pipet 1.0 mL of Molybdate Reagent into each flask.

4. Add 1 Amino Acid pillow to each flask and mix thouroughly. Wait 10 minutes for colour stabilization.

5. Adjust the Wavelength Dial of the spectrophotometer to read 530 nm.

5. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

6. Replace the Zero Scale card with the SODIUM PHOSPHATE in WATER card.

7. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

8. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

9. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Sodium Phosphate concentration from the card and record this result.

Configurations of Sodium Phosphate -

$NaH_2PO_4 \cdot H_2O$	Monobasic
Sodium Phosphate Monohydrate	
Na_2HPO_4	
Dibasic Sodium Phosphate	
$Na_3PO_4 \cdot 12 H_2O$	Tribasic
Sodium Phosphate Dodecahydrate	

SODIUM SULFATE IN SOILS

THORIN TITRIMETRIC TEST

Reference: Environment Canada, Analytical Methods Manual, 1974.

Range: 0-25000 mg/kg

Common Synonyms: Disodium Sulfate, Salt Cake, Glauber's Salt

HAZARDOUS DATA

Human Health
Symptoms: Ingestion - low toxicity. Contact - skin- not irritating, but prolonged contact with concentrated solutions or powders should be avoided; eyes- mechanical irritation and watering.
Toxicology: Low toxicity by all routes.

EXPERIMENTAL REQUIREMENTS

Apparatus: 1 x 100 mL Graduated Cylinder
2 x 250 mL Erlenmeyer Flasks
1 x 125 mL Erlenmeyer Flask fitted with a bored #6 Rubber Stopper
2 x Long-Stem Funnels
Whatman 12.5 cm #1 Filter Paper
1 x 25 mL Burette
1 x 1000 mL Volumetric Flask
5 x 10 mL Disposable Pipets
6 x 150 mL Beakers
125 mL Separatory Funnel
Magnetic Stir Bars
Battery Operated Magnetic Stirrer
Glass Wool
Handpump
pH Meter & Electrode

REQUIRED REAGENTS

20% Hydrochloric Acid solution
1% Hydrochloric Acid solution
1% Ammonium Hydroxide solution
0.00164 N Barium Chloride solution
Ion - Exchange Resin (Dowex 50W-X8)
Thorin indicator solution
Ethanol

Preparation of 20% Hydrochloric Acid solution -

To a 400 mL beaker containing 200 mL of deionized water, slowly add 50 mL of concentrated hydrochloric acid.

Preparation of 1% Hydrochloric Acid -

To a 100 mL beaker containing 95 mL of deionized water, slowly add 5 mL of the 20% hydrochloric acid solution.

Preparation of 1% Ammonium Hydroxide solution -

To a 100 mL beaker containing 99 mL of deionized water, add 1 mL of concentrated ammonium hydroxide.

Preparation of 0.00164 N Barium Chloride solution -

Dissolve 0.400 g of Barium Chloride in a 500 mL beaker containing 400 mL of deionized water. Adjust the solution to pH 3.9 by adding 1% hydrochloric acid solution dropwise. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water.

Preparation of Ion-Exchange Resin -

Insert a small glass wool plug into the neck of a 125 mL separatory funnel. Add 25 g of Dowex 50W-X8 cation exchange resin.

Preparation of Thorin indicator -

Dissolve 0.2 g of Thorin in deionized water in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Prepare the ion-exchange column (as illustrated) by passing 25 mL of 20% hydrochloric acid through it. Discard the spent acid.

6. Pass 2 x 20 mL aliquots of deionized water through the ion-exchange column. Discard the spent water.

7. Pass 20 mL of the BLANK solution through the ion-exchange column. Discard the spent solution.

8. Pass a 30 mL aliquot of the BLANK solution through the ion-exchange column. Pipet 10 mL of this into a clean 150 mL beaker.

9. Repeat steps 5 through 8 using the TEST sample in place of the BLANK sample.

10. Using the graduated cylinder, add 40 mL of Ethanol to each beaker.

11. Adjust both solutions to pH 3.8-4.0 using 1% hydrochloric acid or 1% ammonium hydroxide as required.

12. Add 2-3 drops of thorin indicator to each beaker. The solution should now be orange in colour.

13. Fill the 0-25 mL burette with the barium chloride solution. Record the initial burette reading.

14. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

15. Titrate the BLANK solution by slowly adding the barium chloride until the first permanent pink colour appears. Record the final volume.

16. Repeat step 15 with the TEST solution, making sure to record both initial and final burette readings.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Aluminum Sulfate by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x 225]

Other Sodium Sulfate compounds - (next page)

$Na_2(SO_4)$ - Concentration (mg/kg) = [a.t.v. x 225]

$Na_2(SO_4).7\ H_2O$ - Concentration (mg/kg) = [a.t.v. x 440]

$Na_2(SO_4).10\ H_2O$ - Concentration (mg/kg) = [a.t.v. x 530]

SULFURIC ACID IN WATER

SODIUM HYDROXIDE TITRATION METHOD

Reference: Hach Water Analysis Handbook, Hach Company, 1981.

Range: 0 - 10000 mg/L

Common Synonyms: Hydrogen Sulfate, Battery Acid, Dipping Acid.

HAZARDOUS DATA

Human Health
Symptoms: Highly concentrated acid is rapidly destructive to body tissue on contact. Contact - skin- dermatitis and burns; eyes- rapidly causes severe damage, possible loss of sight. Inhalation - damage to upper respiratory tract, coughing, laboured breathing. Ingestion - sore throat, abdominal pain, nausea, vomiting.
Toxicology: TLV - 1 mg/cubic meter

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 50 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
1 x 25 mL Burette

REQUIRED REAGENTS

Phenolphthalein indicator solution
0.02N Sodium Hydroxide solution
*Note: All required reagents supplied by Hach company

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Fill the 0 - 25 mL burette with 0.02N Sodium Hydroxide solution and record the initial burette reading.

4. Add 6 drops of phenolphthalein solution to each flask.

5. Titrate the BLANK water sample against the sodium hydroxide solution; add slowly, with continuous swirling until a pink colour appears for more than 30 seconds.

6. Record the final burette reading.

7. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Sulfuric Acid by solving the following equation:

$$\text{Concentration (mg/L)} = [\ (\text{adjusted titrant volume}) \times 19.5\ ]$$

para-TERPHENYL IN SOILS

TETRACYANOETHYLENE COLORIMETRIC TEST

Reference: Schinck, G.H., Ozolins, M., Complexometric Determination of Aromatic Hydrocarbons with Tetracyanoethylene, Anal. Chem., 33, 11, 1961.

Range: 0-15000 mg/kg

Common Synonyms: Diphenylbenzene.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - burning sensation, lung damage. Contact - skin and eyes- irritating. Ingestion - burning sensation in throat.
Toxicology: Moderately toxic by ingestion and inhalation.
TLV - 0.05 ppm; 5 mg/cubic meter

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 250 mL Erlenmeyer Flasks
2 x 150 mL Beakers
2 x 50 mL Stoppered Graduated Mixing Cylinders
2 x 25 mL Graduated Cylinders
1 x 200 mL Volumetric Flask
5 x 5 mL Disposable Pipets
1 x 100 mL Beaker
Timing Device
Whatman #1 Filter Paper
2 x Long-stemmed Funnels
Balance

REQUIRED REAGENTS

0.1 M Tetracyanoethylene Solution
Chloroform

Preparation of 0.1 M Tetracyanoethylene solution -

Dissolve 2.562 g of tetracyanoethylene in 10 mL of acetone in a

100 mL beaker. Transfer the solution to a 200 mL volumetric flask with methylene chloride. Dilute to the 200 mL mark with methylene chloride. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Transfer 25 mL of each filtrate to separate 50 mL mixing cylinders.

6. Pipet 5 mL of the tetracyanoethylene solution into each mixing cylinder, stopper, and mix well. Wait 2-3 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the spectrophotometer to read 550 nm.

5. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

6. Replace the Zero Scale card with the p-TERPHENYL in SOIL card.

7. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

8. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

9. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the p-Terphenyl in Soil concentration from the card and record this result.

TOLUENE IN SOILS

TETRACYANOETHYLENE COLORIMETRIC TEST

Reference: Schinck, G.H., Ozolins, M., Complexometric Determination of Aromatic Hydrocarbons with Tetracyanoethylene, Anal. Chem., 33, 11, 1961.

Range: 0-10000 mg/kg

Common Synonyms: Toluol, Methyl Benzene, Methyl Benzol, Phenyl Methane.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - headache, nausea, loss of appetite, rapidly developing pulmonary edema. Contact - skin- may be absorbed; eyes- irritation. Ingestion - vomiting, nausea, irritability, central nervous system depression, depression of bone marrow, depressed respiration.
Toxicology: Moderately toxic by all routes.
TLV - (skin) 100 ppm; 375 mg/cubic meter
Short-term Inhalation Limits - (skin) 150 ppm; 560 mg/cubic meter

Environment:
Aquatic Toxicity Rating - 10 to 100 ppm/96h/TLm/freshwater;

EXPERIMENTAL REQUIREMENTS

Apparatus:
- Hach Spectrophotometer and 2 Cuvettes
- 2 x 250 mL Erlenmeyer Flasks
- 2 x 150 mL Beakers
- 2 x 50 mL Stoppered Graduated Mixing Cylinders
- 2 x 25 mL Graduated Cylinders
- 1 x 200 mL Volumetric Flask
- 5 x 5 mL Disposable Pipets
- 1 x 100 mL Beaker
- Timing Device
- Whatman #1 Filter Paper
- 2 x Long-stemmed Funnels
- Balance

REQUIRED REAGENTS

0.1 M Tetracyanoethylene Solution
Chloroform

Preparation of 0.1 M Tetracyanoethylene solution -

Dissolve 2.562 g of tetracyanoethylene in 10 mL of acetone in a 100 mL beaker. Transfer the solution to a 200 mL volumetric flask with methylene chloride. Dilute to the 200 mL mark with methylene chloride. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of chloroform to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Transfer 25 mL of each filtrate to separate 50 mL mixing cylinders.

6. Pipet 5 mL of the tetracyanoethylene solution into each mixing cylinder, stopper, and mix well. Wait 2-3 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the spectrophotometer to read 440 nm.

8. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

9. Replace the Zero Scale card with the TOLUENE in SOIL card.

10. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

11. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

12. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Toluene in Soil concentration from the card and record this result.

TOLUENE IN WATER AND WASTEWATER

TETRACYANOETHYLENE COLORIMETRIC TEST

Reference: Schinck, G.H., Ozolins, M., Complexometric Determination of Aromatic Hydrocarbons with Tetracyanoethylene, Anal. Chem., 33, 11, 1961.

Range: 0-2500 mg/L

Common Synonyms: Toluol, Methyl Benzene, Methyl Benzol, Phenyl Methane.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - headache, nausea, loss of appetite, rapidly developing pulmonary edema. Contact - skin- may be absorbed; eyes- irritation. Ingestion - vomiting, nausea, irritability, central nervous system depression, depression of bone marrow, depressed respiration.
Toxicology: Moderately toxic by all routes.
TLV - (skin) 100 ppm; 375 mg/cubic meter
Short-term Inhalation Limits - (skin) 150 ppm; 560 mg/cubic meter

Environment:
Aquatic Toxicity Rating - 10 to 100 ppm/96h/TLm/freshwater;

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 150 mL Beakers
2 x 125 mL Separatory Funnels
2 x 50 mL Stoppered Graduated Mixing Cylinders
2 x 25 mL Graduated Cylinders
1 x 200 mL Volumetric Flask
5 x 5 mL Disposable Pipets
1 x 100 mL Beaker
Timing Device
Balance

REQUIRED REAGENTS

0.1 M Tetracyanoethylene Solution
Chloroform

Preparation of 0.1 M Tetracyanoethylene solution -

Dissolve 2.562 g of tetracyanoethylene in 10 mL of acetone in a 100 mL beaker. Transfer the solution to a 200 mL volumetric flask with methylene chloride. Dilute to the 200 mL mark with methylene chloride. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 25 mL water sample in a graduated cylinder. Pour the sample into a clean 125 mL separatory funnel. Label the funnel BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 25 mL mark. Pour the water into a second clean 125 mL separatory funnel. Label the funnel TEST.

3. Pipet 25 mL of chloroform into each of the 125 mL separatory funnels, stopper them, and shake well. Invert the separatory funnel periodically and open the stopcock to vent any buildup of gases.

4. Allow the phases to separate (usually 2-3 minutes).

5. Drain from each funnel 25 mL of the bottom (chloroform) layer into separate graduated mixing cylinders.

6. Pipet 5 mL of the tetracyanoethylene solution into each mixing cylinder, stopper, and mix well. Wait 2-3 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the meter to read 480 nm.

8. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

9. Replace the Zero Scale card with the TOLUENE in WATER card.

10. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

11. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

12. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Toluene in Water concentration from the card and record this result.

UREA IN SOILS

p-DIMETHYLAMINOBENZALDEHYDE COLORIMETRIC TEST

Reference: Watt, G.W., Chrisp, J.D., Anal. Chem., 26, 3, (1954)

Range: 0 - 4500 mg/kg

Common Synonyms: Carbamide, Carbonyldiamide.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - (dust) sore throat, coughing, shortness of breath. Contact - skin or eyes- causes redness and irritation. Ingestion - sore throat, abdominal pain.
Toxicology: Moderately toxic by inhalation and contact.

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
2 x 100 mL Mixing Cylinders
2 x 150 mL beakers
2 x 5 mL Graduated Disposable Pipets
Whatman #1 Filter Paper
2 x Long-Stemmed Funnels
Balance

REQUIRED REAGENTS

p-Dimethylaminobenzaldehyde solution

Preparation of p-Dimethylaminobenzaldehyde solution -

Dissolve 5.000 g of p-dimethylaminobenzaldehyde in 50 mL of ethanol in a 100 mL beaker. Acidify the solution by slowly adding 15 mL of concentrated hydrochloric acid. Transfer the solution to a 250 mL volumetric flask and dilute to the 250 mL mark with ethanol. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Pour 50 mL of each of the blank and contaminated filtrates into separate clean 100 mL mixing cylinders.

6. Pipet 5.0 mL of the p-Dimethylaminobenzaldehyde solution into each mixing cylinder. Stopper, mix and wait 2 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the spectrophotometer to read 425 nm.

8. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

9. Replace the Zero Scale card with the UREA in SOIL card.

10. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

11. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

12. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Urea concentration from the card and record this result.

UREA IN WATER AND WASTEWATER

p-DIMETHYLAMINOBENZALDEHYDE COLORIMETRIC TEST

Reference: Watt, G.W., Chrisp, J.D., Anal. Chem., 26, 3, (1954)

Range: 0 - 200 mg/L

Common Synonyms: Carbamide, Carbonyldiamide.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - (dust) sore throat, coughing, shortness of breath. Contact - skin or eyes- causes redness and irritation. Ingestion - sore throat, abdominal pain.
Toxicology: Moderately toxic by inhalation and contact.

Environment:
Aquatic Toxicity Rating - > 1000 ppm/96h/TLm/freshwater
Drinking Water: Maximum allowable concentration in Class I waters is 10 mg/L (Verschueren, 1984).

EXPERIMENTAL REQUIREMENTS

Apparatus: Hach Spectrophotometer and 2 Cuvettes
2 x 100 mL Graduated Cylinders
2 x 250 mL Erlenmeyer Flasks
2 x 10 mL Graduated Disposable Pipets

REQUIRED REAGENTS

p-Dimethylaminobenzaldehyde solution

Preparation of p-Dimethylaminobenzaldehyde solution -

Dissolve 5.000 g of p-dimethylaminobenzaldehyde in 50 mL of ethanol in a 100 mL beaker. Acidify the solution by slowly adding 15 mL of concentrated hydrochloric acid. Transfer the solution to a 250 mL volumetric flask and dilute to the 250 mL mark with ethanol. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 25 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 25 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Pipet 10.0 mL of the p-Dimethylaminobenzaldehyde solution into each flask. Swirl the contents and wait 2 minutes for colour stabilization.

4. Adjust the Wavelength Dial of the spectrophotometer to read 425 nm.

5. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

6. Replace the Zero Scale card with the UREA in WATER card.

7. Fill one cuvette to the 25 mL mark from the flask labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

8. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

9. Fill the other cuvette to the 25 mL mark from the flask labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Urea concentration from the card and record this result.

VINYL ACETATE IN SOIL

POTASSIUM HYDROXIDE TITRATION METHOD

Reference: Critchfield, F.E., Organic Functional Group Analysis, Pergamon Press, New York, 1963.

Range: 250 - 3000 mg/kg

Common Synonyms: Acetic Acid Ethenyl Ester, Acetic Acid Vinyl Ester, VAC.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - anaesthetic effect. Ingestion - nausea and vomiting. Contact - skin and eyes - liquid and vapours irritating.
Toxicology: Moderately toxic by all routes.

Environment:
Aquatic Toxicity Rating - 10 to 100/96h/TLm/freshwater

EXPERIMENTAL REQUIREMENTS

Apparatus: 2 x 250 mL Erlenmeyer Flasks
2 x Funnels
Whatman #1 Filter Paper
2 x 250 mL Beakers
50 mL Graduated Cylinder
25 mL Burette
2 x Magnetic Stirring Bars
Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

0.12M Mercuric Acetate solution
0.1N Potassium Hydroxide solution
Sodium Bromide Packet (4 grams)
Phenolphthalein indicator solution

Preparation of Mercuric Acetate solution, 0.12M -

Dissolve 7.648 grams of Mercuric Acetate in 100 mL of Methanol in a 150 mL beaker. Transfer this solution to a 200 mL volumetric flask and dilute to the mark with Methanol.

Preparation of Potassium Hydroxide solution, 0.1N -

Dissolve 2.806 grams of Potassium Hydroxide in 150 mL of Methanol in a 250 mL beaker. Transfer this solution to a 500 mL volumetric flask and dilute to the mark with Methanol.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 250 mL beakers. Label the beakers to match the originating flask.

5. Put a magnetic stirring bar in each beaker.

6. Add 50 mL of Mercuric Acetate solution to each beaker. Allow 10 minutes for the reaction to proceed.

7. Add a packet of sodium bromide and 5 drops of the phenolphthalein indicator solution to each beaker.

8. Fill the burette with 0.1N Potassium Hydroxide. Record the initial burette reading. Place the BLANK sample on the magnetic stirrer. Turn stirrer to low speed. Titrate the BLANK sample with Potassium Hydroxide until the first permanent pink colour. Record final burette reading.

9. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Vinyl Acetate by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x (titrant concentration) x 7400]

where titrant concentration is 0.1 unless standard Potassium Hydroxide solution is not used.

XYLENES IN SOILS

TETRACYANOETHYLENE COLORIMETRIC TEST

Reference: Schinck, G.H., Ozolins, M., Complexometric Determination of Aromatic Hydrocarbons with Tetracyanoethylene, Anal. Chem., 33, 11, 1961.

Range: 0-7500 mg/kg

Common Synonyms: Xylols, 1,2- , 1,3- , 1,4- Dimethylbenzene, o- , m- , p- dimethylbenzene.

Xylenes are typically a mixture of the three isomers, ortho, meta, para, with the last two being the predominant portion.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - dizziness, coughing, nausea, vomiting, fatigue, drowsiness, narcosis. Contact - skin- dryness, cracking; eyes- redness, watering, inflammation. Ingestion - gastrointestinal irritation, dizziness, fatigue, loss of consciousness.
Toxicology: Moderately toxic by all routes.
TLV - (skin) 100 ppm; 435 mg/cubic meter
Short-term Inhalation Limits - (skin) 150 ppm; 655 mg/cubic meter (15 min)

Environment:
Aquatic Toxicity Rating - 10 to 100 ppm/96h/TLm/freshwater;

EXPERIMENTAL REQUIREMENTS

Apparatus:
- Hach Spectrophotometer and 2 Cuvettes
- 2 x 250 mL Erlenmeyer Flasks
- 2 x 150 mL Beakers
- 2 x 50 mL Stoppered Graduated Mixing Cylinders
- 2 x 25 mL Graduated Cylinders
- 1 x 200 mL Volumetric Flask
- 5 x 5 mL Disposable Pipets
- 1 x 100 mL Beaker
- Timing Device
- Whatman #1 Filter Paper
- 2 x Long-stemmed Funnels
- Balance

REQUIRED REAGENTS

0.1 M Tetracyanoethylene Solution
Chloroform

Preparation of 0.1 M Tetracyanoethylene solution -

Dissolve 2.562 g of tetracyanoethylene in 10 mL of acetone in a 100 mL beaker. Transfer the solution to a 200 mL volumetric flask with methylene chloride. Dilute to the 200 mL mark with methylene chloride. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 50 mL of chloroform to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Transfer 25 mL of each filtrate to separate 50 mL mixing cylinders.

6. Pipet 5 mL of the tetracyanoethylene solution into each mixing cylinder, stopper, and mix well. Wait 2-3 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the spectrophotometer to read 440 nm.

8. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

9. Replace the Zero Scale card with the the appropriate XYLENE in SOIL card (if a xylene mixture, use the meta-XYLENE in SOIL card).

10. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the

spectrophotometer and close the lid.

11. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

12. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Xylene in Soil concentration from the card and record this result.

XYLENES IN WATER AND WASTEWATER

TETRACYANOETHYLENE COLORIMETRIC TEST

Reference: Schinck, G.H., Ozolins, M., Complexometric Determination of Aromatic Hydrocarbons with Tetracyanoethylene, Anal. Chem., 33, 11, 1961.

Range: 0 - 1500 mg/L

Common Synonyms: Xylols, 1,2- , 1,3- , 1,4- Dimethylbenzene, o- , m- , p- dimethylbenzene.

Xylenes are typically a mixture of the three isomers, ortho, meta, para, with the last two being the predominant portion.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - dizziness, coughing, nausea, vomiting, fatigue, drowsiness, narcosis. Contact - skin- dryness, cracking; eyes- redness, watering, inflammation. Ingestion - gastrointestinal irritation, dizziness, fatigue, loss of consciousness.
Toxicology: Moderately toxic by all routes.
TLV - (skin) 100 ppm; 435 mg/cubic meter
Short-term Inhalation Limits - (skin) 150 ppm; 655 mg/cubic meter (15 min)

Environment:
Aquatic Toxicity Rating - 10 to 100 ppm/96h/TLm/freshwater;

EXPERIMENTAL REQUIREMENTS

Apparatus:
- Hach Spectrophotometer and 2 Cuvettes
- 2 x 150 mL Beakers
- 2 x 125 mL Separatory Funnels
- 2 x 50 mL Stoppered Graduated Mixing Cylinders
- 2 x 25 mL Graduated Cylinders
- 1 x 200 mL Volumetric Flask
- 5 x 5 mL Disposable Pipets
- 1 x 100 mL Beaker
- Timing Device
- Balance

REQUIRED REAGENTS

0.1 M Tetracyanoethylene Solution
Chloroform

Preparation of 0.1 M Tetracyanoethylene solution -

Dissolve 2.562 g of tetracyanoethylene in 10 mL of acetone in a 100 mL beaker. Transfer the solution to a 200 mL volumetric flask with methylene chloride. Dilute to the 200 mL mark with methylene chloride. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 25 mL water sample in a graduated cylinder. Pour the sample into a clean 125 mL separatory funnel. Label the funnel BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 25 mL mark. Pour the water into a second clean 125 mL separatory funnel. Label the funnel TEST.

3. Pipet 25 mL of chloroform into each of the 125 mL separatory funnels, stopper them, and shake well. Invert the separatory funnel periodically and open the stopcock to vent any buildup of gases.

4. Allow the phases to separate (usually 2-3 minutes).

5. Drain from each funnel 25 mL of the bottom (chloroform) layer into separate graduated mixing cylinders.

6. Pipet 5 mL of the tetracyanoethylene solution into each mixing cylinder, stopper, and mix well. Wait 2-3 minutes for colour stabilization.

7. Adjust the Wavelength Dial of the spectrophotometer to read 480 nm.

8. Insert the Zero Scale card into the card holder of the spectrophotometer. Hold the toggle switch to Zero Check and adjust the Zero Adjust Dial until the needle position corresponds to the zero mark on the card.

9. Replace the Zero Scale card with the the appropriate XYLENE in WATER card (if a xylene mixture, use the meta-XYLENE in WATER card).

10. Fill one cuvette to the 25 mL mark from the beaker labelled BLANK. Insert this cuvette into the sample holder of the spectrophotometer and close the lid.

11. Turn the toggle switch to ON. Turn the Light Control Dial until the needle position corresponds to the zero mark on the card. Turn the switch to OFF and remove the sample.

12. Fill the other cuvette to the 25 mL mark from the beaker labelled TEST. Insert this cuvette into the sample holder and close the lid. Turn the toggle switch to ON. Read the Xylene in Water concentration from the card and record this result.

ZINC CHLORIDE IN SOILS

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/kg

Common Synonyms: Butter of Zinc, Zinc Dichloride, Zinc Muriate Solution

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of nose and throat, headache, cough, chest pain, fever, nausea. Ingestion - swallowing large amounts may cause abdominal pains, nausea, diarrhea, shock. Contact - may cause severe skin irritation and serious eye injury.
Toxicology: Highly toxic by inhalation. Moderately toxic by ingestion and contact.
TLV - (inhalation) 1 mg/cubic meter (as Zinc Chloride fume)
Short-term Inhalation Limit - 2 mg/cubic meter (15 min)

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 2 x Long-Stem Funnels
- Whatman 12.5 cm #1 Filter Paper
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 1 x 100 mL Volumetric Flask
- 2 x 50 mL Beakers
- 4 x 150 mL Beakers
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer
- pH Meter and Electrode

REQUIRED REAGENTS

- 0.1 N Nitric Acid solution
- 0.014 N Mercuric Nitrate solution
- Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to match the originating flask.

5. Pipet 25 mL of each filtrate into two more separate, clean 150 mL beakers. Add 75 mL of deionized water to each using the graduated cylinder.

6. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

7. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

8. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

9. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

10. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

11. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Zinc Chloride by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x 384]

ZINC CHLORIDE IN WATER AND WASTEWATER

MERCURIC NITRATE TITRIMETRIC TEST

Reference: Standard Methods for the Examination of Water and Wastewater, Fourteenth Ed., 1975.

Range: 0-25000 mg/L

Common Synonyms: Butter of Zinc, Zinc Dichloride, Zinc Muriate Solution

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - irritation of nose and throat, headache, cough, chest pain, fever, nausea. Ingestion - swallowing large amounts may cause abdominal pains, nausea, diarrhea, shock. Contact - may cause severe skin irritation and serious eye injury.
Toxicology: Highly toxic by inhalation. Moderately toxic by ingestion and contact.
TLV - (inhalation) 1 mg/cubic meter (as Zinc Chloride fume)
Short-term Inhalation Limit - 2 mg/cubic meter (15 min)

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 1 x 100 mL Volumetric Flask
- 2 x 50 mL Beakers
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer

REQUIRED REAGENTS

- 0.1 N Nitric Acid solution
- 0.014 N Mercuric Nitrate solution
- Chloride indicator solution

Preparation of 0.1 N Nitric Acid solution -

Pour approximately 1 litre of deionized water into a clean glass-stoppered bottle. With a graduated cylinder or pipet, add 6.5 mL of concentrated nitric acid. Stopper the bottle, mix well, and label the bottle.

Preparation of 0.014 N Mercuric Nitrate solution -

Dissolve 2.415 g of Mercuric Nitrate Hydrate in 100 mL of deionized water in a 150 mL beaker. With a graduated cylinder or pipet, add 2 mL of concentrated Nitric Acid. Transfer the solution to a 1000 mL volumetric flask and dilute to the mark with deionized water. Mix thoroughly.

Preparation of Chloride indicator solution -

Dissolve 0.250 g of s-Diphenylcarbazone and 0.030 g of Xylene Cyanol FF in methanol in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with methanol. Mix thoroughly.

PROCEDURE

1. From an uncontaminated source in the area obtain a 50 mL water sample in a graduated cylinder. Pour the sample into a clean 250 mL Erlenmeyer flask. Label the flask BLANK.

2. Take a water sample from the contaminated area of interest by filling a clean graduated cylinder to the 50 mL mark. Pour the water into a second clean 250 mL Erlenmeyer flask. Label the flask TEST.

3. Using a calibrated electrode, adjust the pH of each solution to pH 2.5 by adding 0.1N Nitric acid dropwise.

4. Add approximately 1 mL of the chloride indicator solution to each beaker (the solutions should now be blue).

5. Fill the 0-25 mL burette with the 0.014 N mercuric nitrate solution. Record the initial burette reading.

6. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

7. Titrate the BLANK solution by slowly adding the mercuric nitrate until the first permanent purple colour appears. Record the final volume.

8. Refill the burette. Record initial burette reading. Titrate

the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Zinc Chloride by solving the following equation:

Concentration (mg/L) = [(adjusted titrant volume) x 19.21]

ZINC SULFATE IN SOILS

THORIN TITRIMETRIC TEST

Reference: Environment Canada, Analytical Methods Manual, 1974.

Range: 0-25000 mg/kg

Common Synonyms: Zinc Sulfate Monohydrate, Zinc Sulfate Heptahydrate, White Vitriol, White Copperas.

HAZARDOUS DATA

Human Health
Symptoms: Inhalation - sore throat, coughing, shortness of breath, laboured breathing. Ingestion - abdominal spasms, diarrhea, vomiting. Contact - skin and eyes - redness and pain.
Toxicology: Moderately toxic by inhalation and contact.

EXPERIMENTAL REQUIREMENTS

Apparatus:
- 1 x 100 mL Graduated Cylinder
- 2 x 250 mL Erlenmeyer Flasks
- 1 x 125 mL Erlenmeyer Flask fitted with a bored #6 Rubber Stopper
- 2 x Long-Stem Funnels
- Whatman 12.5 cm #1 Filter Paper
- 1 x 25 mL Burette
- 1 x 1000 mL Volumetric Flask
- 5 x 10 mL Disposable Pipets
- 6 x 150 mL Beakers
- 125 mL Separatory Funnel
- Magnetic Stir Bars
- Battery Operated Magnetic Stirrer
- Glass Wool
- Handpump
- pH Meter & Electrode

REQUIRED REAGENTS

- 20% Hydrochloric Acid solution
- 1% Hydrochloric Acid solution
- 1% Ammonium Hydroxide solution
- 0.00164 N Barium Chloride solution
- Ion - Exchange Resin (Dowex 50W-X8)
- Thorin indicator solution

Ethanol

Preparation of 20% Hydrochloric Acid solution -

To a 400 mL beaker containing 200 mL of deionized water, slowly add 50 mL of concentrated hydrochloric acid.

Preparation of 1% Hydrochloric Acid -

To a 100 mL beaker containing 95 mL of deionized water, slowly add 5 mL of the 20% hydrochloric acid solution.

Preparation of 1% Ammonium Hydroxide solution -

To a 100 mL beaker containing 99 mL of deionized water, add 1 mL of concentrated ammonium hydroxide.

Preparation of 0.00164 N Barium Chloride solution -

Dissolve 0.400 g of Barium Chloride in a 500 mL beaker containing 400 mL of deionized water. Adjust the solution to pH 3.9 by adding 1% hydrochloric acid solution dropwise. Transfer the solution to a 1000 mL volumetric flask and dilute to the 1000 mL mark with deionized water.

Preparation of Ion-Exchange Resin -

Insert a small glass wool plug into the neck of a 125 mL separatory funnel. Add 25 g of Dowex 50W-X8 cation exchange resin.

Preparation of Thorin indicator -

Dissolve 0.2 g of Thorin in deionized water in a 50 mL beaker. Transfer the solution to a 100 mL volumetric flask and dilute to the 100 mL mark with deionized water.

PROCEDURE

1. Weigh into a clean 250 mL Erlenmeyer flask 10 grams of soil from an uncontaminated area. Label the flask BLANK.

2. Weigh into a second clean Erlenmeyer flask 10 grams of soil from the contaminated area of interest. Label the flask TEST.

3. Using a graduated cylinder, transfer 100 mL of water to each flask. Mix well.

4. Filter the contents of each flask through Whatman #1 filter paper into separate, clean 150 mL beakers. Label the beakers to

match the originating flask.

5. Prepare the ion-exchange column (as illustrated) by passing 25 mL of 20% hydrochloric acid through it. Discard the spent acid.

6. Pass 2 x 20 mL aliquots of deionized water through the ion-exchange column. Discard the spent water.

7. Pass 20 mL of the BLANK solution through the ion-exchange column. Discard the spent solution.

8. Pass a 30 mL aliquot of the BLANK solution through the ion-exchange column. Pipet 10 mL of this into a clean 150 mL beaker.

9. Repeat steps 5 through 8 using the TEST sample in place of the BLANK sample.

10. Using the graduated cylinder, add 40 mL of Ethanol to each beaker.

11. Adjust both solutions to pH 3.8-4.0 using 1% hydrochloric acid or 1% ammonium hydroxide as required.

12. Add 2-3 drops of thorin indicator to each beaker. The solution should now be orange in colour.

13. Fill the 0-25 mL burette with the barium chloride solution. Record the initial burette reading.

14. Add a magnetic stir bar to each beaker, place the BLANK solution on the magnetic stirrer and turn ON.

15. Titrate the BLANK solution by slowly adding the barium chloride until the first permanent pink colour appears. Record the final volume.

16. Refill the burette. Record initial burette reading. Titrate the TEST sample as above. Record final burette reading.

CALCULATIONS

1. Calculate the Titrant Volume by subtracting the initial burette reading from the final reading for both the BLANK and TEST samples.

2. Subtract the BLANK titrant volume from the TEST titrant volume to find the adjusted titrant volume (in mLs).

3. Calculate the concentration of Zinc Sulfate by solving the following equation:

Concentration (mg/kg) = [(adjusted titrant volume) x 265]

Other Zinc Sulfate compounds -

$Zn(SO4)$ - Concentration (mg/kg) = [a.t.v. x 265]

$Zn(SO4).\ 6\ H2O$ - Concentration (mg/kg) = [a.t.v. x 443]

$Zn(SO4).\ 7\ H2O$ - Concentration (mg/kg) = [a.t.v. x 496]

SECTION B

ACETONE

WATER		SOIL	
CONCENTRATION (mg/L)	ABSORBANCE	CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0	0.0	0.0
10	0.045	50	0.185
25	0.115	100	0.275
50	0.32	200	0.468
100	0.475	300	0.603
200	0.67	400	0.700
400	0.94	500	0.765

AMMONIUM PHOSPHATE DIBASIC

WATER

CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0
2.0	0.057
4.0	0.114
6.0	0.172
8.0	0.235
10	0.293
15	0.450
20	0.600
25	0.752
30	0.905

AMMONIUM PHOSPHATE MONOBASIC

WATER

CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0
1.0	0.04
2.0	0.08
3.0	0.119
4.0	0.159
5.0	0.199
10	0.398
15	0.597
20	0.796
25	0.994

BENZENE

WATER

CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0
500	0.043
1000	0.079
2000	0.149
4000	0.29
5000	0.36
6000	0.423
7000	0.494
8000	0.571
9000	0.642
10000	0.712

SOIL

CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0
1000	0.035
2000	0.069
3000	0.104
4000	0.138
5000	0.173
7000	0.242
9000	0.311
10000	0.346

iso-BUTYRALDEHYDE

WATER

CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0
5.0	0.12
10	0.185
15	0.263
20	0.312
25	0.337
30	0.355
35	0.37
40	0.38

TYPE: SANDY SOIL

CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0
25	0.065
50	0.171
75	0.276
100	0.382
125	0.487
150	0.592
175	0.698
200	0.803

TYPE: CLAY BASED SOIL

CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0
25	0.03
50	0.06
75	0.09
100	0.12
125	0.15
150	0.18
175	0.21
200	0.24
300	0.36
400	0.48
500	0.60
600	0.72

n-BUTYRALDEHYDE

WATER		SOIL	
CONCENTRATION (mg/L)	ABSORBANCE	CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0	0.0	0.0
1.0	0.05	25	0.101
2.0	0.10	50	0.252
3.0	0.175	75	0.404
4.0	0.25	100	0.555
5.0	0.30	125	0.706
10	0.57	150	0.857
15	0.67		
20	0.75		
25	0.81		
30	0.87		
35	0.91		
40	0.94		

CALCIUM PHOSPHATE MONOBASIC MONOHYDRATE

WATER	
CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0
1.0	0.038
2.0	0.075
3.0	0.113
4.0	0.151
5.0	0.189
10	0.377
15	0.566
20	0.754
25	0.942

CUMENE

WATER

CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0
250	0.03
500	0.067
1000	0.14
1500	0.21
2000	0.288
2500	0.362
3000	0.436
3500	0.51
4000	0.58

SOIL

CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0
500	0.028
1000	0.056
1500	0.084
2000	0.112
3000	0.167
4000	0.223
5000	0.279
6000	0.335
7000	0.391
8000	0.446
9000	0.502
10000	0.558

ETHYLBENZENE

WATER

CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0
250	0.046
500	0.11
750	0.173
1000	0.236
1500	0.363
2000	0.489
2500	0.616
3000	0.869

SOIL

CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0
500	0.051
1000	0.102
2000	0.205
3000	0.307
4000	0.410
5000	0.512
6000	0.615
7000	0.717
8000	0.82

HYDROGEN PEROXIDE

WATER

CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0
1.0	0.079
2.0	0.126
3.0	0.178
4.0	0.234
5.0	0.288
7.0	0.395
10	0.558
15	0.828

METHYL ETHYL KETONE

WATER		SOIL	
CONCENTRATION (mg/L)	ABSORBANCE	CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0	0.0	0.0
25	0.075	50	0.054
50	0.15	100	0.107
100	0.26	150	0.161
200	0.425	200	0.214
300	0.54	250	0.268
400	0.625	300	0.322
		400	0.429
		500	0.536

METHYL ISOBUTYL KETONE

WATER		SOIL	
CONCENTRATION (mg/L)	ABSORBANCE	CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0	0.0	0.0
25	0.045	50	0.077
50	0.08	100	0.153
100	0.15	200	0.306
200	0.27	300	0.459
300	0.44	400	0.612
		500	0.765

PHOSPHORIC ACID

WATER	
CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0
1.0	0.076
2.0	0.153
3.0	0.230
4.0	0.307
6.0	0.460
8.0	0.613
10	0.767
12	0.920

p-TERPHENYL

SOIL

CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0
1000	0.023
2000	0.046
3000	0.069
4000	0.092
5000	0.116
6000	0.139
7000	0.162
8000	0.185
9000	0.208
10000	0.231
15000	0.347

TOLUENE

WATER

CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0
250	0.0545
500	0.134
750	0.213
1000	0.292
1500	0.45
2000	0.609
2500	0.768

SOIL

CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0
500	0.019
1000	0.045
2000	0.095
3000	0.145
4000	0.195
5000	0.245
6000	0.296
7000	0.346
8000	0.397
9000	0.447
10000	0.497

UREA

WATER

CONCENTRATION (mg/L)	ABSORBANCE
0.0	0.0
25	0.05
50	0.090
100	0.16
150	0.225
200	0.27

SOIL

CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0
500	0.084
1000	0.142
2000	0.26
3000	0.376
4500	0.55

m-XYLENE

WATER		SOIL	
CONCENTRATION (mg/L)	ABSORBANCE	CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0	0.0	0.0
100	0.051	500	0.067
200	0.139	1000	0.134
300	0.228	1500	0.202
400	0.317	2000	0.269
500	0.406	3000	0.403
600	0.494	4000	0.538
700	0.583	5000	0.672
800	0.672	6000	0.806
900	0.76	7000	0.941
1000	0.85		

o-XYLENE

WATER		SOIL	
CONCENTRATION (mg/L)	ABSORBANCE	CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0	0.0	0.0
100	0.045	250	0.036
200	0.115	500	0.072
300	0.185	1000	0.143
400	0.255	1500	0.215
500	0.326	2000	0.286
600	0.396	3000	0.429
700	0.466	4000	0.572
800	0.536	5000	0.716
900	0.606	6000	1.002
1000	0.676		
1500	1.02		

p-XYLENE

WATER		SOIL	
CONCENTRATION (mg/L)	ABSORBANCE	CONCENTRATION (mg/kg)	ABSORBANCE
0.0	0.0	0.0	0.0
100	0.055	500	0.061
200	0.099	1000	0.118
300	0.153	1500	0.169
400	0.20	2000	0.219
500	0.25	3000	0.32
600	0.30	4000	0.421
700	0.35	5000	0.522
800	0.398	6000	0.623
900	0.447		
1000	0.496		
1250	0.62		
1500	0.74		

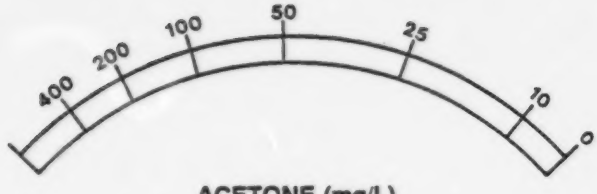
SECTION C

Acetone in Soil

ACETONE (mg/kg)
2,4-Dinitrophenylhydrazine Test
480 nm

Environment Canada Environnement Canada

Acetone in Water



ACETONE (mg/L)
2,4-Dinitrophenylhydrazine Test
480 nm

Environment Canada Environnement Canada

Ammonium Nitrate in Soil

AMMONIUM NITRATE
(mg/kg)
Cadmium Reduction Test
500 nm

Environment Canada Environnement Canada

Amonium Nitrate in Water

AMMONIUM NITRATE
(mg/L)
Cadmium Reduction Test
500 nm

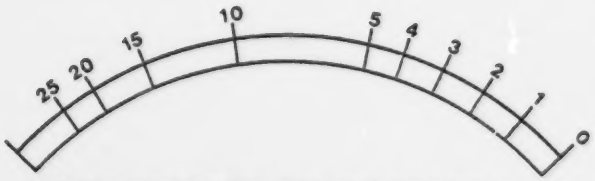
Environment Canada Environnement Canada

$(NH_4)_2HPO_4$ In Water

AMMONIUM PHOSPHATE, DIBASIC (mg/L)
Amino Acid Test
530 nm

Environment Canada Environnement Canada

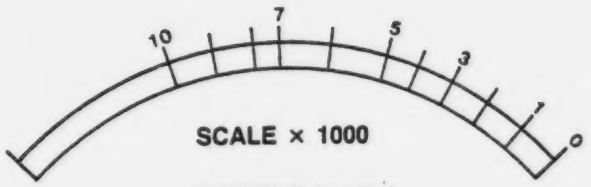
$NH_4H_2PO_4$ in Water



AMMONIUM PHOSPHATE, MONOBASIC (mg/L)
Amino Acid Test
530 nm

Environment Canada Environnement Canada

Benzene In Soil



SCALE × 1000

BENZENE (mg/kg)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

Benzene In Water

SCALE × 1000

BENZENE (mg/L)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

1-Butanol In Soil

SCALE × 1000

1-BUTANOL (mg/kg)
Ceric Ammonium Nitrate Test
450 nm

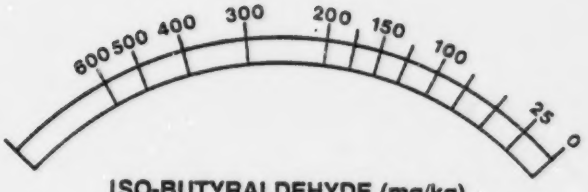
Environment Canada Environnement Canada

1-Butanol in Water

1-BUTANOL (mg/L)
Ceric Ammonium Nitrate Test
450 nm

Environment Canada Environnement Canada

Iso-Butyraldehyde in Clay Based Soil



ISO-BUTYRALDEHYDE (mg/kg)
2,4-Dinitrophenylhydrazine Test
480 nm

Environment Canada Environnement Canada

Iso-Butyraldehyde in Sandy Soil

ISO-BUTYRALDEHYDE (mg/kg)
2,4-Dinitrophenylhydrazine Test
480 nm

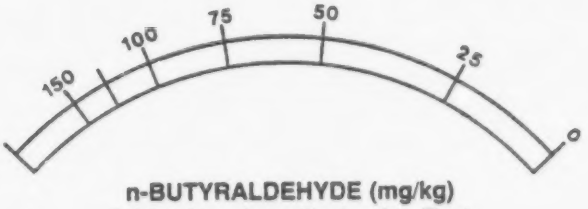
Environment Canada Environnement Canada

iso-Butyraldehyde in Water

ISO-BUTYRALDEHYDE (mg/L)
2,4-Dinitrophenylhydrazine Test
480 nm

Environment Canada Environnement Canada

n-Butyraldehyde in Soil



n-BUTYRALDEHYDE (mg/kg)
2,4-Dinitrophenylhydrazine Test
480 nm

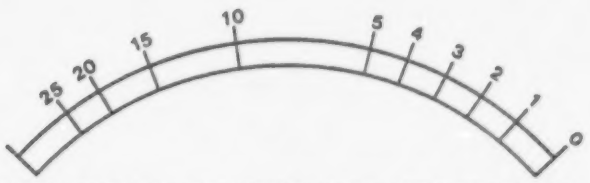
Environment Canada Environnement Canada

n-Butyraldehyde in Water

n-BUTYRALDEHYDE (mg/L)
2,4-Dinitrophenylhydrazine Test
480 nm

Environment Canada Environnement Canada

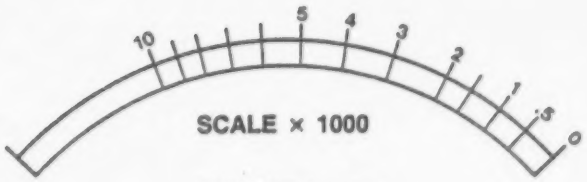
$Ca(H_2PO_4)_2 \cdot H_2O$ in Water



CALCIUM PHOSPHATE, MONOBASIC, MONOHYDRATE
(mg/L)
Amino Acid Test
530 nm

Environment Canada Environnement Canada

Cumene in Soil

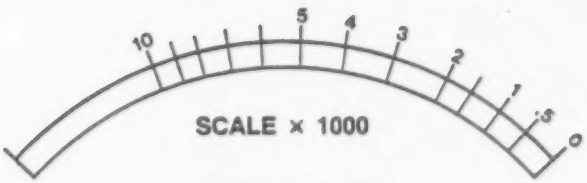


SCALE × 1000

CUMENE (mg/kg)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

Cumene in Soil



SCALE × 1000

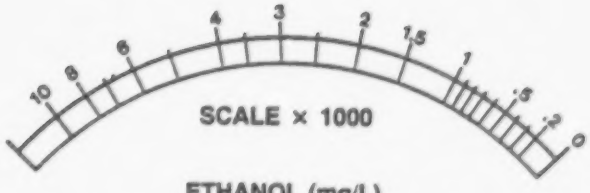
CUMENE (mg/kg)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

Ethanol in Soil

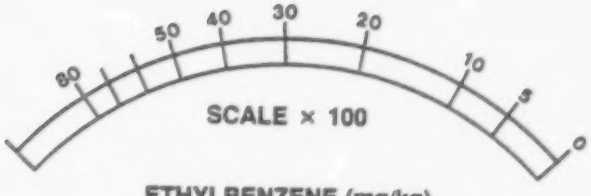
ETHANOL (mg/kg)
Ceric Ammonium Nitrate Test
450 nm

Ethanol in Water



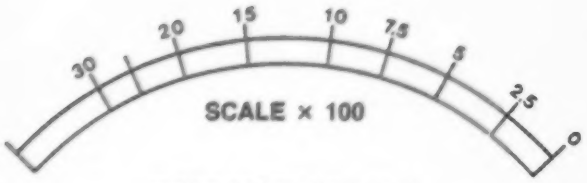
ETHANOL (mg/L)
Ceric Ammonium Nitrate Test
450 nm

Ethylbenzene in Soil



ETHYLBENZENE (mg/kg)
Tetracyanoethylene Test
440 nm

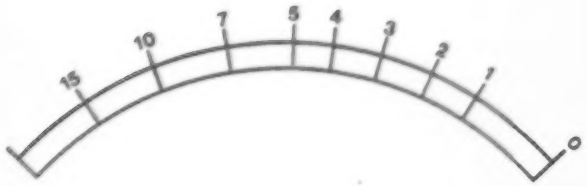
Ethylbenzene in Water



ETHYLBENZENE (mg/L)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

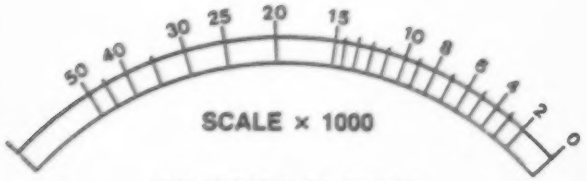
Hydrogen Peroxide in Water



HYDROGEN PEROXIDE (mg/L)
o-Tolidine Test
450 nm

Environment Canada Environnement Canada

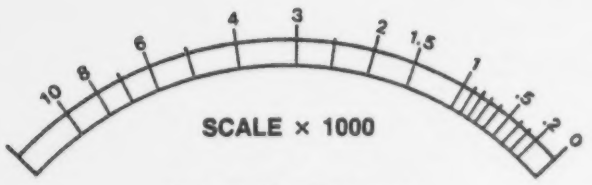
Iso-Propanol in Soil



ISO-PROPANOL (mg/kg)
Ceric Ammonium Nitrate Test
455 nm

Environment Canada Environnement Canada

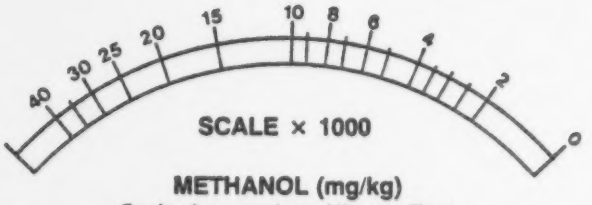
Iso-Propanol in Water



ISO-PROPANOL (mg/L)
Ceric Ammonium Nitrate Test
450 nm

Environment Canada Environnement Canada

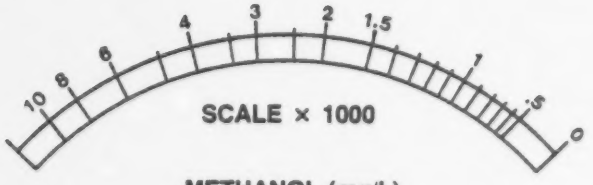
Methanol in Soil



METHANOL (mg/kg)
Ceric Ammonium Nitrate Test
450 nm

Environment Canada Environnement Canada

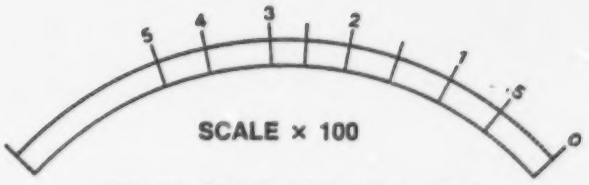
Methanol in Water



METHANOL (mg/L)
Ceric Ammonium Nitrate Test
460 nm

Environment Canada Environnement Canada

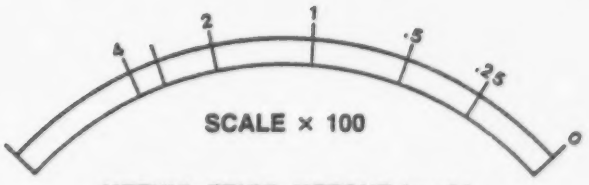
MEK in Soil



METHYL ETHYL KETONE (mg/kg)
2,4-Dinitrophenylhydrazine Test
480 nm

Environment Canada Environnement Canada

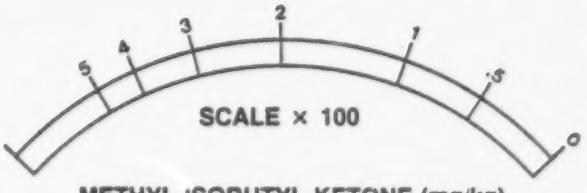
MEK in Water



METHYL ETHYL KETONE (mg/L)
2,4-Dinitrophenylhydrazine Test
480 nm

Environment Canada Environnement Canada

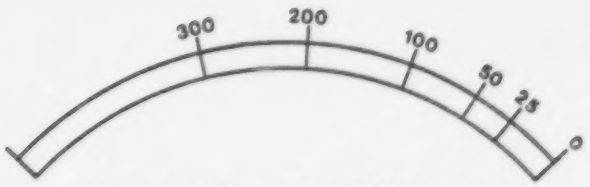
MIBK in Soil



METHYL ISOBUTYL KETONE (mg/kg)
2,4-Dinitrophenylhydrazine Test
480 nm

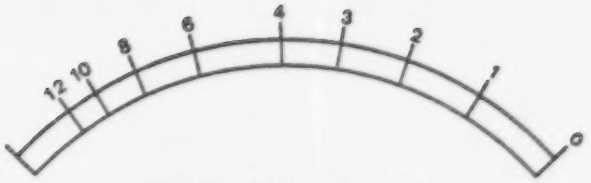
Environment Canada Environnement Canada

MIBK in Water



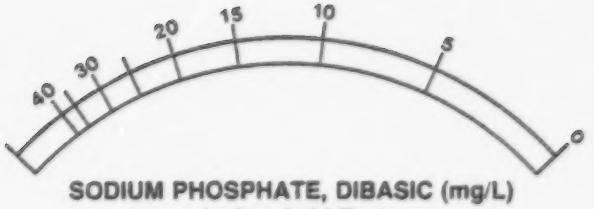
METHYL ISOBUTYL KETONE (mg/L)
2,4-Dinitrophenylhydrazine Test
480 nm

H_3PO_4 in Water



PHOSPHORIC ACID (mg/L)
Amino Acid Test
530 nm

Na_2HPO_4 In Water



SODIUM PHOSPHATE, DIBASIC (mg/L)
Amino Acid Test
530 nm

$NaH_2PO_4.H_2O$ in Water

SODIUM PHOSPHATE, MONOBASIC, MONOHYDRATE (mg/L)
Amino Acid Test
530 nm

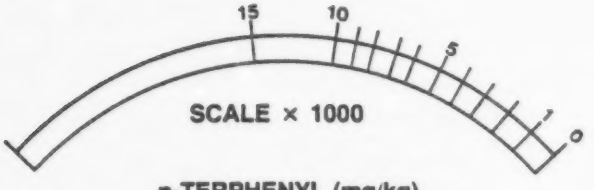
Environment Canada Environnement Canada

$Na_3PO_4.12H_2O$ in Water

SODIUM PHOSPHATE, TRIBASIC DODECAHYDRATE (mg/L)
Amino Acid Test
530 nm

Environment Canada Environnement Canada

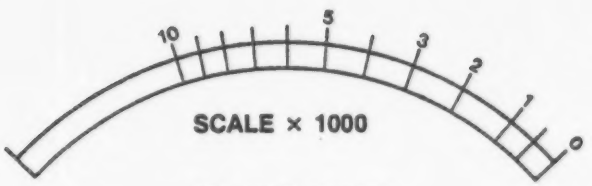
p-Terphenyl in Soil



p-TERPHENYL (mg/kg)
Tetracyanoethylene Test
550 nm

Environment Canada Environnement Canada

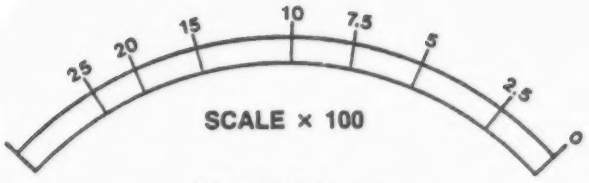
Toluene in Soil



TOLUENE (mg/kg)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

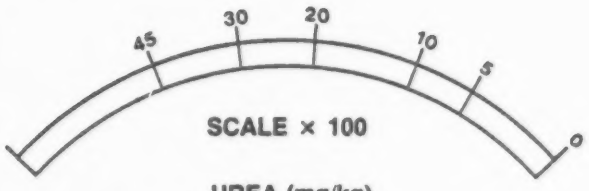
Toluene in Water



TOLUENE (mg/L)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

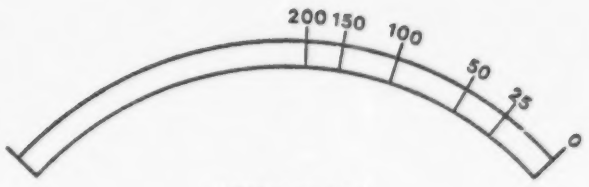
Urea in Soil



UREA (mg/kg)
p-Dimethylaminobenzaldehyde Test
425 nm

Environment Canada Environnement Canada

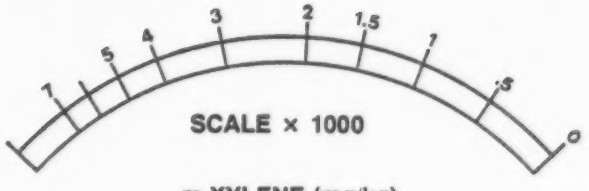
Urea in Water



UREA (mg/L)
p-Dimethylaminobenzaldehyde Test
425 nm

Environment Canada Environnement Canada

m-Xylene in Soil

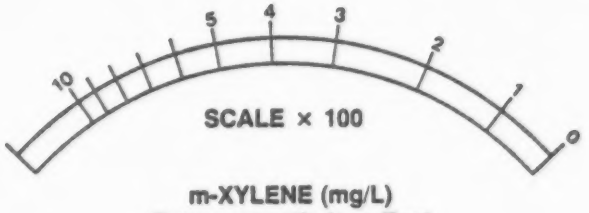


SCALE × 1000

m-XYLENE (mg/kg)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

m-Xylene in Water

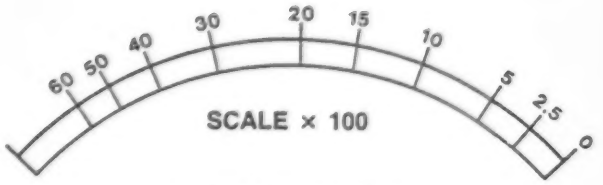


SCALE × 100

m-XYLENE (mg/L)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

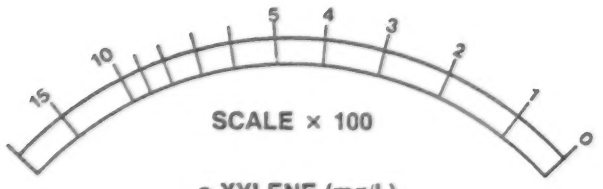
o-Xylene in Soil



o-XYLENE (mg/kg)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

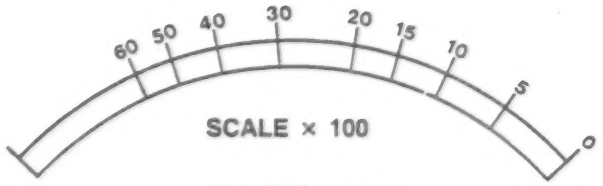
o-Xylene in Water



o-XYLENE (mg/L)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

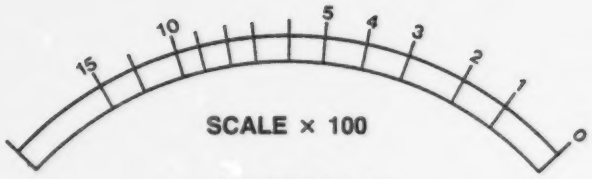
p-Xylene in Soil



p-XYLENE (mg/kg)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

p-Xylene in Water



SCALE × 100

p-XYLENE (mg/L)
Tetracyanoethylene Test
440 nm

Environment Canada Environnement Canada

SECTION D

SUMMARY OF PPAK CAPABILITIES

in water-

ANALYTE	RANGE (mg/L)	TYPE OF TEST
Acetic Acid	0-10000	Titrimetric
Acetic Anhydride	10-2000	Titrimetric
Acetone	0-400	Colorimetric
Acrylonitrile	100-2000	Titrimetric
Adipic Acid	0-10000	Titrimetric
Aluminum Chloride	0-25000	Titrimetric
-Hexahydrate	0-25000	Titrimetric
Ammonium Chloride	0-25000	Titrimetric
Ammonium Nitrate	0-80	Colorimetric
Ammonium Phosphate-		
Dibasic	0-30	Colorimetric
Monobasic	0-25	Colorimetric
Benzene	0-15000	Colorimetric
Benzoic Acid	0-10000	Titrimetric
1-Butanol	200-8000	Colorimetric
iso-Butyraldehyde	0-40	Colorimetric
n-Butyraldehyde	0-40	Colorimetric
Calcium Chloride	0-25000	Titrimetric
-Monohydrate	0-25000	Titrimetric
-Dihydrate	0-25000	Titrimetric
-Hexahydrate	0-25000	Titrimetric
Calcium Hydroxide	0-10000	Titrimetric
Calcium Oxide	0-10000	Titrimetric
Calcium Phosphate	0-25	Colorimetric
*Carbon Dioxide	0-1000	Titrimetric
*Chlorine, Free	0-2	Colorimetric
*Chlorine, Total	0-2	Titrimetric
Cumene	0-15000	Colorimetric
Ethanol	200-10000	Colorimetric
Ethanolamine	0-500	Titrimetric
Ethylbenzene	0-750	Colorimetric
Ethylene Glycol	0-500	Titrimetric
Ferric Chloride	0-25000	Titrimetric
-2.5 Hydrate	0-25000	Titrimetric
-Hexahydrate	0-25000	Titrimetric
Formic Acid	0-10000	Titrimetric
Hydrochloric Acid	0-25000	Titrimetric
Hydrochloric Acid	0-10000	Titrimetric
Hydrogen Peroxide	0-10	Colorimetric
Hydrogen Peroxide	0-75	Titrimetric
Isopropanol	200-10000	Colorimetric
Maleic Anhydride	25-1000	Titrimetric
*Mercury	0-100	Colorimetric
Methyl Ethyl Ketone	0-400	Colorimetric
Methanol	500-10000	Colorimetric
MIBK	0-400	Colorimetric
Morpholine	100-2000	Titrimetric

Nitric Acid	0-10000	Titrimetric
NTA	40-1500	Titrimetric
*Phenol	0-0.2	Colorimetric
Phosphoric Acid	0-15	Colorimetric
Phosphoric Acid	0-10000	Titrimetric
*Phosphorus	0-20	Colorimetric
Potassium Chloride	0-25000	Titrimetric
Potassium Hydroxide	0-10000	Titrimetric
Propylene Glycol	0-500	Titrimetric
Sodium Carbonate	0-10000	Titrimetric
Sodium Chloride	0-25000	Titrimetric
Sodium Hydroxide	0-10000	Titrimetric
Sodium Phosphates	0-70	Colorimetric
Sulfuric Acid	0-10000	Titrimetric
Toluene	0-2500	Colorimetric
Urea	0-200	Colorimetric
o-Xylene	0-1500	Colorimetric
m-Xylene	0-1000	Colorimetric
p-Xylene	0-1500	Colorimetric
Zinc Chloride	0-25000	Titrimetric

* designates those tests which were already included in the HACH kit.

in soil-

ANALYTE	RANGE (mg/kg)	TYPE OF TEST
Acetic Anhydride	500-5000	Titrimetric
Acrylonitrile	500-4000	Titrimetric
Adipic Acid	200-4000	Titrimetric
Aluminum Chloride	0-25000	Titrimetric
-Hexahydrate	0-25000	Titrimetric
Aluminum Sulfate	0-25000	Titrimetric
-18 Hydrate	0-25000	Titrimetric
Ammonium Chloride	0-25000	Titrimetric
Ammonium Nitrate	100-4000	Colorimetric
Ammonium Sulfate	0-25000	Titrimetric
Benzene	0-15000	Colorimetric
Boric Acid	500-5000	Titrimetric
1-Butanol	2000-40000	Colorimetric
iso-Butyraldehyde	0-150	Colorimetric
n-Butyraldehyde	0-200	Colorimetric
Calcium Chloride	0-25000	Titrimetric
-Monohydrate	0-25000	Titrimetric
-Dihydrate	0-25000	Titrimetric
-Hexahydrate	0-25000	Titrimetric
Cumene	0-15000	Colorimetric
Ethanol	2000-35000	Colorimetric
Ethylbenzene	0-10000	Colorimetric
Ethylene Glycol	100-3000	Titrimetric
Ferric Chloride	0-25000	Titrimetric
-2.5 Hydrate	0-25000	Titrimetric
-Hexahydrate	0-25000	Titrimetric

Hydrochloric Acid	0-25000	Titrimetric
Isopropanol	2000-50000	Colorimetric
Maleic Anhydride	250-5000	Titrimetric
Methyl Ethyl Ketone	0-500	Colorimetric
Methanol	2000-40000	Colorimetric
MIBK	0-500	Colorimetric
Morpholine	500-5000	Titrimetric
NTA	250-5000	Titrimetric
Phthalic Anhydride	200-4000	Titrimetric
Potassium Chloride	0-25000	Titrimetric
Potassium Sulfate	0-25000	Titrimetric
Propylene Glycol	150-3000	Titrimetric
Sodium Chloride	0-25000	Titrimetric
Sodium Sulfate	0-25000	Titrimetric
-Heptahydrate	0-25000	Titrimetric
-Decahydrate	0-25000	Titrimetric
p-Terphenyl	0-15000	Colorimetric
Toluene	0-10000	Colorimetric
Urea	0-4500	Colorimetric
Vinyl Acetate	250-3000	Titrimetric
o-Xylene	0-6000	Colorimetric
m-Xylene	0-7000	Colorimetric
p-Xylene	0-6000	Colorimetric
Zinc Chloride	0-25000	Titrimetric
Zinc Sulfate	0-25000	Titrimetric
-Hexahydrate	0-25000	Titrimetric
-Heptahydrate	0-25000	Titrimetric

SECTION E

PPAK IMPLEMENTATION REQUIREMENTS

In addition to the Hach kit, the following items would be required for full implementation of the PPAK.

Additional Reagents Required:

Ceric Ammonium Nitrate
Acetic Anhydride
Acrylonitrile
Morpholine
Tetracyanoethylene
o-Tolidine
Sodium Acetate
Ethanol
p-Dimethylaminobenzaldehyde
Sodium Chloride
Barium Chloride
Anhydrous Alcohol
Mercuric Nitrate
Naphthalene
Sodium Metal
Sodium Phosphate
Mercuric Acetate
Sodium Bromide

Solvents:
- Methanol
- Ethyl Cellosolve
- Methyl Cellosolve
- Pyridine
- Methylene Chloride
- Carbon Tetrachloride
- Ether

Standard Solutions:
- 0.100 M Iodine
- 0.100 M Arsenious Acid
- 0.100 M Perchloric Acid

Indicators:
- Thorin
- Thymol Blue/ Xylene Cyanol FF
- S-Diphenyl Carbazone/ Xylene Cyanol FF

Additional Equipment Required:

Cole-Parmer Portable Magnetic Stirrer
Magnetic Stirring Bars
Standard 25 mL Burette
2 x 125 mL Separatory Funnels
2 x 250 mL Erlenmeyer Flasks
2 x Long-Stemmed Funnels
Filter Paper
2 x 150 mL Beaker
Replace Hach pH meter with Presto-Tek DspH-1 portable pH meter.